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The Journal *of the* Society of Dyers and Colourists

Volume 77 Number 4

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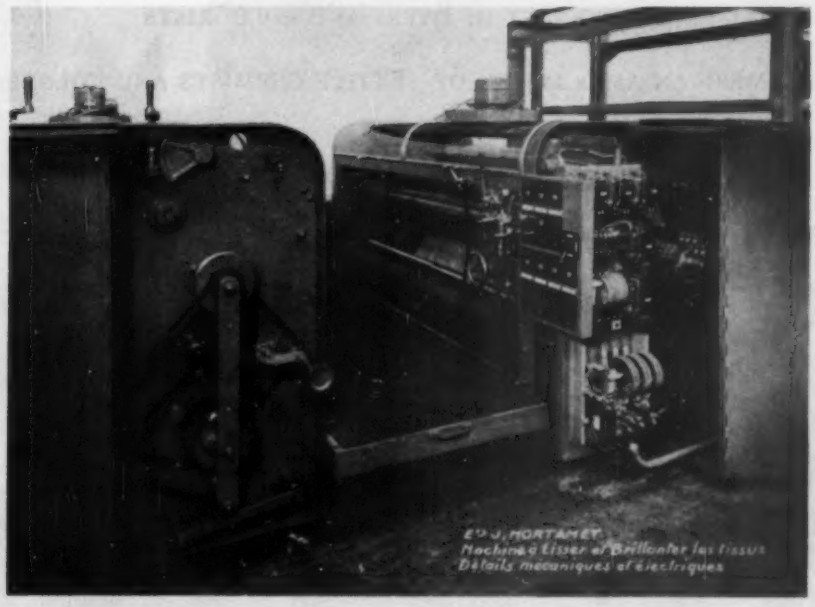

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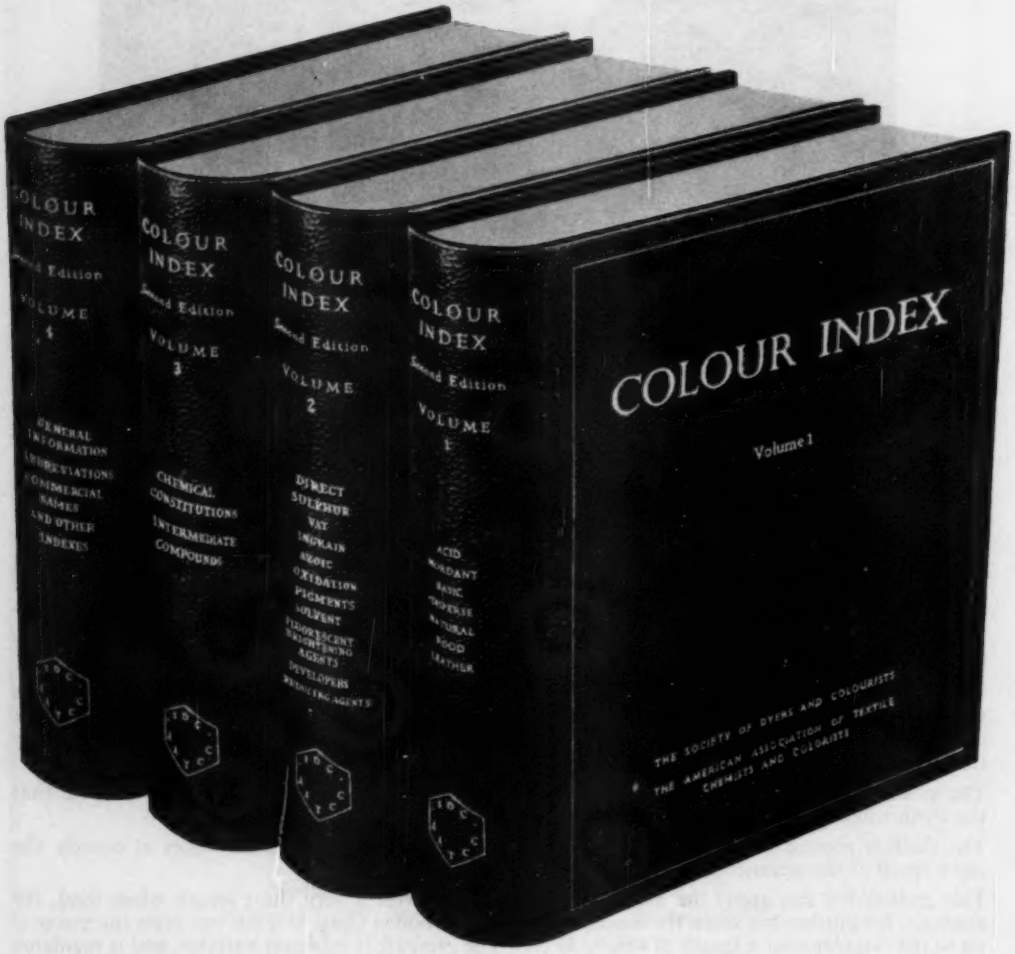

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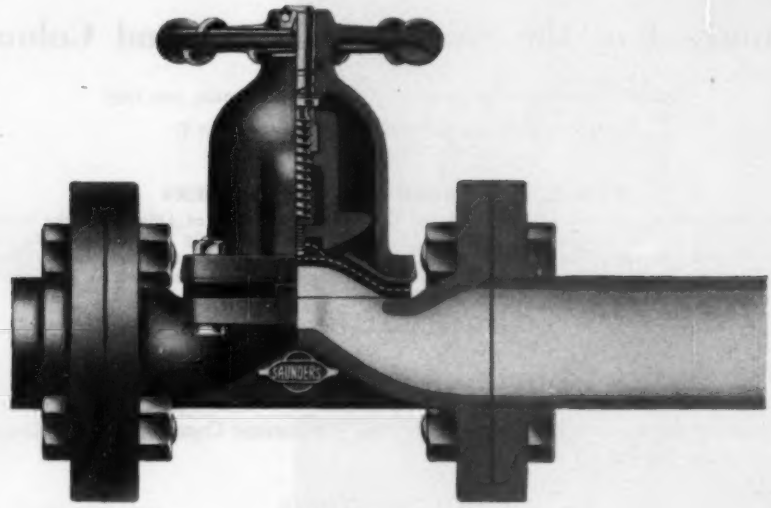

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(Abstracts section only—see January issue, page 2)

NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–8 of the January 1961 and pages 389–396 of the July 1960 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

Action of Tetrakis(hydroxymethyl)phosphonium Chloride on Wool and Hair — *L. S. Bajpai, C. S. Whewell, and J. M. Woodhouse*

Foam, Flow, and Level Dyeing (Some New Information on Factors Influencing the Flow of Dye Liquor through Wool Packages) — *H. R. Hadfield and D. R. Lemin*

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Volume 77 Number 4 APRIL 1961 Issued Monthly

COMMUNICATIONS

Setting of Wool Fibres in Dyeing

R. V. Peryman and R. F. Pickup

The degree of permanence of set (to release in boiling water) in single wool fibres which have been dyed whilst held stretched, varies widely with the method of dyeing.

Dyeing under neutral to slightly alkaline conditions and dyeing by afterchrome methods produce high degrees of permanent set, but supercontraction can occur after the use of strongly acid or acid chrome-mordant methods with sexivalent chromium. All dyeing processes give temporary set, but this can be much less with certain processes carried out at low temperature or under strongly acid conditions. The steaming of tufts of fibres, previously dyed (or chrome-dyed) by different methods under tension, produces marked differences in length and crimp. Twist is set more completely than extension and is less affected by subsequent steaming.

Introduction

The study of the setting of keratin fibres continues to receive attention because of the bearing of the physical and chemical mechanism on fibre structure [1–9] and because of the practical importance of producing permanent set [10, 11]. Although references have been made [12, 13] to the ability of processed fibres to take set, the setting of keratin fibres during dyeing does not appear to have been considered. In the work now described, the setting effect of different dyeing processes has been studied and some observations regarding the chemical mechanisms are made.

Mechanical processing tends to introduce into fibres strain which is modified in subsequent wet processing. Thus in dyeing, the strains in some fibres will be released, but other fibres, with less freedom of movement, will remain strained. Fibres may also become strained during the dyeing process itself, either by the flow of liquor in a circulating machine or by the pull on a piece running on a winch. Fibres in a state of strain during dyeing will be subject to set in varying degrees. Recently Gullbrandson [14] has drawn attention to the weakening effect of kinks set in fibres during dyeing of loose wool. Subsequent treatment at the boil, e.g. re-dyeing or steaming of the dyed material, may release at least some of the set produced in the original dyeing and, under certain conditions, supercontraction may occur.

Experimental

The general plan of the investigation was to determine the degree of set retained by the root halves of four or more stretched wool fibres after dyeing by various methods and after subsequent release for 15 min and 60 min in boiling water. In the light of the results obtained, methods of dyeing giving widely different degrees of permanent set were applied to (*a*) bundles of fibres subsequently cut to simulate tufts, and (*b*) bundles of fibres twisted to simulate yarn.

SINGLE FIBRES

(*a*) *Material*

Root halves of staples selected visually for uniformity from 50–56s New Zealand Romney crossbred fleece wool were extracted with light petroleum, washed once with cold ethanol, then 6 times with distilled water at 40°C, and allowed to dry in air.

(*b*) *Procedure*

Four fibres, selected visually, each about 7·5 cm long, were mounted individually in a four-fibre stretching frame, stretched 30% taking 5–10 min in water at room temperature, and after about 30 min treated (see Tables I–III) in a Lister laboratory dyeing machine [15], a 3·5-l. lading can, a Longclose laboratory package-dyeing machine, or a Thies high-temperature dyeing machine. Most treatments, except those at high temperatures, were repeated in different machines. The liquor ratio of 50:1 was achieved by including tubular knitted fabric of 64s quality together with a smaller amount of 56s quality for the determination of chromium content. The liquor ratio of 100:1 in the high-temperature treatments was obtained by similar means. Details of treatments are given in Tables I–III; the fibres and fabric were introduced at 50°C and the liquor was brought to the boil in 45 min for the boiling treatments or brought to the higher temperatures in 30 min. The tensioned fibres were slackened in the early stages of the first of two 10-min rinses at 40–45°C immediately following the dyeing treatment. After the treated

TABLE I

Set Produced by Dyeing and Related Treatments excluding Chroming and Treatments above 100°C
(Single fibres, extension 30%)

No.	Treatment	Set (%)† After "dyeing"	Set (%)† After subsequent boiling in water for 15 min	Set (%)† After subsequent boiling in water for 60 min
	Boiled 1½ h at pH 1·8–2·9 using H_2SO_4*			
1	Blank with 10% Na_2SO_4 and acid for pH 2·4	12·1	−5·1	−6·3
2	6% Neolan Blue 2G (C.I. Acid Blue 158A), 7% Na_2SO_4, 8% H_2SO_4, at pH 1·8	11·0	−4·4	−6·8
3	6% Disulphine Blue FFS (C.I. Acid Blue 147), 5% Na_2SO_4, 5% H_2SO_4, for 1 h at pH 2·0	14·0	−7·8	−9·7
4	8% Neopalatine Dark Blue G (C.I. Acid Black 8), 3% Uniperol W (BASF), 3% H_2SO_4	20·1	−2·3	−5·9
	Boiled 1½ h at pH 3·4–3·5 using formic acid*			
5	Blank with 8% Na_2SO_4 and acid	22·4	0·4	0·1
6	8% Wool Red 10B (see C.I. Acid Violet 5), 5% Na_2SO_4	17·0	1·5	−1·4
7	0·5% formaldehyde, 8% Na_2SO_4	18·9	−0·1	−3·0
8	1·3% formaldehyde, 8% Na_2SO_4	22·2	—	−7·6
9	2·6% formaldehyde, 8% Na_2SO_4	19·3	−8·9	−14·6
	Treatments with stripping agents using 4% formic acid (90%)			
10	4% Formosul (C.I. Reducing Agent 2) for 50 min at 85–95°C	22·5	—	19·6
11	4% Vacolite (C.I. Reducing Agent 6) for 50 min at 85–95°C	25·4	20·2	18·6
12	4% Neovadine AN (Ciba) for 1½ h at boil	22·7	10·3	7·9
	Boiled ½ h at pH 3·6–3·7 using formic acid*			
13	(*a*) Blank with acid	20·7	0·9	−0·7
13	(*b*) 5% Procion Yellow RS (C.I. Reactive Yellow 4)	20·3	3·8	0·4
	Boiled 1½ h at pH 4–5 using acetic acid*			
14	Blank with 4% Na_2SO_4 and acid for pH 4·8	25·0	16·5	12·0
15	Coomassie Blue BL (C.I. Acid Blue 59), 4% Na_2SO_4 and acid for pH 4·5	26·5	18·8	14·8
	Boiled 1½ h at pH 5·7–8·2 using ammonium acetate*			
16	5% Coomassie Navy Blue 2RNS (C.I. Acid Blue 113), 5% ammonium acetate, at pH 7·8–5·9, adjusting with NH_4OH	25·7	—	20·9
17	5% Polar Red 3B conc. (C.I. Acid Red 134), 5% ammonium acetate, at pH 8·2–5·7, adjusting with NH_4OH	25·6	—	21·2
	*Boiled 1½ h with ammonium sulphate at pH 7·5–8·6**			
18	8% Cibalan Black BGL (C.I. Acid Black 107), 4% ammonium sulphate, at pH 7·5–7·9, adjusting with NH_4OH	26·0	19·7	17·6
19	8% Carbolan Blue BS (C.I. Acid Blue 138), 4% ammonium sulphate, at pH 8·6, adjusting with NH_4OH	24·9	22·1	20·2
	Vat dyeing with Indigo [22]			
20	Indigo pdr. (C.I. Vat Blue 1) with weakly alkaline hydrosulphite, 3 dips	23·9	−2·7	−6·6
21	As above and then top with 3% Erio Fast Purple A (Gy), 5% Na_2SO_4, 5% acetic acid (30%) and boil 1 h	22·9	—	16·3
	Dyeing with the aid of a solvent [16]			
22	(*a*) 4% Irgalan Grey BL (C.I. Acid Black 58), 3% ammonium sulphate in satd. aq. solution of n-butyl alcohol at 60°C for 40 min	2·7	−4·4	−5·2
22	(*b*) As above but without the n-butyl alcohol	1·0	0·0	−0·2

* The pH values are of solutions cooled to 20°C.

† Supercontraction is indicated by the negative sign.

fibres had been dried in the air of the laboratory for about 18 h, their lengths were re-measured. The permanence of the set in the fibre to release in boiling water for 60 min was then tested, and in many instances a test for 15-min immersion was also included.

Using the above procedure, stretched fibres were subjected to various treatments simulating practical methods of dyeing, particularly those likely to cause different degrees of set. The subsequent release of the fibres immersed in boiling water for 15 min and 60 min was intended to simulate release given in commercial finishing and re-dyeing, respectively. The amount of set destroyed by release in boiling water or steam for up to 60 min is regarded as temporary set, whilst that which survives is regarded as permanent set. When the fibre contracts to less than its original length, supercontraction has occurred.

In considering the results the relation [7, 17, 18] between pH and time of setting should be kept in mind: thus, usually, treatment for 1½ h at the boil tends to give permanent set at all but the lowest pH values.

TABLE II

Set produced by Chrome Dyeing and Chroming at the Boil

(Single fibres, extension 30%)

No.	Treatment	Set (%)† After "dyeing"	Set (%)† After subsequent boiling in water for 15 min	Set (%)† After subsequent boiling in water for 60 min	Cr as $K_2Cr_2O_7$ on dry wt. (%)
	Boiled 1½ h or (45 + 45) min at pH 2·4 using H_2SO_4*				
23	Chrome only with 2½% $K_2Cr_2O_7$, 10% Na_2SO_4	10·5	−9·5	−20·5	1·64
24	6% Carmoisine WS (C.I. Acid Red 14), 8% Na_2SO_4, afterchrome with 2½% $K_2Cr_2O_7$	19·3	1·2	−2·1	2·20
	Boiled 1½ h or (45 + 45) min at pH 3·4–3·5 using formic acid*				
25	Chrome only with 2½% $K_2Cr_2O_7$, 6% Na_2SO_4	11·3	−11·0	−23·4	1·96
26	Chrome only with 5% $CrF_3.4H_2O$	25·5	18·1	12·9	1·84
27	Chrome only, afterchrome, ½% $K_2Cr_2O_7$	23·8	—	1·0	0·43
28	Chrome only, afterchrome, 2½% $K_2Cr_2O_7$	23·9	—	2·0	1·22
29	8% Wool Red 10B (see C.I. Acid Violet 5), 5% Na_2SO_4, 1% $K_2Cr_2O_7$	13·4	−4·9	−9·6	0·83
30	Chrome mordant with 1% $K_2Cr_2O_7$ and dye with 8% Solochrome Black WDFA (C.I. Mordant Black 11)	21·9	−2·2	−6·7	1·0
31	Chrome mordant with 3% $K_2Cr_2O_7$, and dye with 8% Solochrome Black WDFA	19·9	−2·4	−8·2	2·58
32	8% Solochrome Black WDFA, 8% Na_2SO_4, afterchrome with ½% $K_2Cr_2O_7$	24·4	—	5·6	0·39
33	As 32, but afterchrome with 1% $K_2Cr_2O_7$	26·3	—	14·8	0·99
34	As 32, but afterchrome with 3% $K_2Cr_2O_7$	25·3	21·8	18·2	2·44
35	3% Solochrome Black WDFA, 8% Na_2SO_4, afterchrome with 1% $K_2Cr_2O_7$	24·4	15·6	8·2	0·88
36	8% Alizarine Red SW (C.I. Mordant Red 3), afterchrome with 3% $CrF_3.4H_2O$	25·1	12·3	8·8	1·47
37	8% Alizarine Red SW, afterchrome with 2½% $K_2Cr_2O_7$	25·7	20·1	17·2	2·12
	Boiled 1½ h or (45 + 45) min at pH 4–5 using acetic acid*				
38	Chrome only with 2½% $K_2Cr_2O_7$	23·0	4·9	−4·3	2·25
39	Chrome only, afterchrome with 2% $K_2Cr_2O_7$	25·9	23·3	20·5	1·18
40	Chrome mordant with 6% $CrF_3.4H_2O$ and dye with 6% Eriochrome Azurol B (C.I. Mordant Blue 1)	25·2	19·2	13·8	1·51
41	6% Eriochrome Azurol B and afterchrome with 2½% $CrF_3.4H_2O$	26·1	22·0	18·5	0·56
42	Chrome mordant with 2% $K_2Cr_2O_7$ and dye with 6% Eriochrome Azurol B	16·9	1·3	−3·1	1·81
43	6% Eriochrome Azurol B and afterchrome with 2% $K_2Cr_2O_7$	25·8	22·5	20·6	1·25
44	Chrome mordant with 2% $K_2Cr_2O_7$ and HCOOH, and dye with 6% Solway Blue Black BS (C.I. Mordant Black 13)	18·0	−1·1	−8·8	1·35
45	8% Solway Blue Black BS, 5% Na_2SO_4 and afterchrome with 3% $K_2Cr_2O_7$	26·1	23·3	19·0	1·63
46	8% Chrome Fast Black A (C.I. Mordant Black 1) and afterchrome with 1% $K_2Cr_2O_7$	25·3	16·2	13·0	0·68
47	As 46 but afterchrome with 2% $K_2Cr_2O_7$	26·1	19·2	15·9	1·25
48	As 46 but afterchrome with 3% $K_2Cr_2O_7$	25·2	17·4	14·0	1·50
	*Boiled 1½ h with 5% ammonium acetate at pH 5–8**				
49	Afterchrome without dye, with 2½% $K_2Cr_2O_7$	26·2	24·2	22·8	2·01
50	5% Coomassie Navy Blue 2RNS (C.I. Acid Blue 113), and ½% $K_2Cr_2O_7$	24·7	—	13·3	0·39
51	Top with 3% Coomassie Navy Blue 2RNS, ½% $K_2Cr_2O_7$, boiling 45 min after dyeing with Indigo as in No. 20	25·0	—	11·0	0·38
	*Boiled 1½ h with ammonium sulphate at pH 8·6–9·0**				
52	Blank metachrome dyeing with 2½% $K_2Cr_2O_7$, 5% ammonium sulphate, and ammonia	25·1	16·7	10·3	1·18
53	2% Metachrome Brown B (C.I. Mordant Brown 48) and 3% metachrome mordant at pH 8·6	24·4	18·2	12·2	0·55
54	8% Metachrome Brown B and 8% metachrome mordant at pH 8·9–9·0	24·4	18·4	13·3	0·68

* The pH values are of solutions cooled to 20°C.

† Supercontraction is indicated by the negative sign.

TABLE III

Set produced by High-temperature Treatments

(Single fibres, extension 30%)

No.	Treatment at 106°C (Liquor ratio 100:1)	Set (%)† After "dyeing"	Set (%)† After subsequent heating in water for 60 min at boil	Set (%)† After subsequent heating in water for 15 min 106°C	Cr as $K_2Cr_2O_7$ on dry weight (%)
	Treatments at pH 3·4–3·5 using formic acid*				
55	Blank with 16·9% Na_2SO_4 for 20 min	26·9	−3·8	−3·0	0
56	Chrome only with 2½% $K_2Cr_2O_7$ and 12% Na_2SO_4 for 20 min	14·3	−8·5	−12·3	1·14
57	Chrome only, afterchrome method, with 16% Na_2SO_4 and acid for 15 min, 2½% $K_2Cr_2O_7$ added and treatment continued for further 15 min	23·6	−0·7	−9·6	0·76
58	8% Wool Red 10B (see C.I. Acid Violet 5), 10% Na_2SO_4, 1% $K_2Cr_2O_7$ for 20 min	22·1	−3·0	−3·7	0·53
59	Chrome mordant with 3% $K_2Cr_2O_7$ and 5% Na_2SO_4 for 20 min, and dye with 8% Solochrome Black WDFA (C.I. Mordant Black 11) in fresh liquor under same conditions	20·6	−4·8	−7·0	1·31
60	8% Solochrome Black WDFA, 16% Na_2SO_4 for 15 min, and afterchrome with 1% $K_2Cr_2O_7$ under similar conditions	26·3	8·4	2·9	0·53
61	Chrome only with 2½% $K_2Cr_2O_7$ and 12% Na_2SO_4 at 112°C for 20 min	21·1	−12·5	−26·7‡	1·23
	*Treatments at pH 4·4–8·9**				
62	5% Coomassie Blue BL (C.I. Acid Blue 59), 7½% Na_2SO_4 and acetic acid for pH 4·5, for 20 min	27·2	11·3	2·6	0
63	6% Solochrome Brown MS (C.I. Mordant Brown 12), 6% metachrome mordant and ammonia for pH 8·6–8·9 for 20 min	26·9	15·0	3·3	0·24
64	6% Eriochrome Azurol B (C.I. Mordant Blue 1) with acetic acid for pH 4·4 for 15 min, afterchromed with 2% $K_2Cr_2O_7$ similarly	25·5	18·2	7·5	0·94

* The pH values are of solutions cooled to 20°C.

† Supercontraction is indicated by the negative sign.

‡ Heated at 112°C for 15 min.

(*c*) *Total Set or Set after Dyeing*

The results given in Tables I–III have, with one exception, the common feature that considerable total set was produced, the amount depending to some extent on the conditions used. The exception (No. 22a) was the treatment at the low temperature (60°C) by a solvent-assisted method [16], though the similar amount of set produced by the treatment (No. 22b) without the special solvent indicates that temperature is the major factor; in the other treatments strongly acid conditions, with or without chromic acid (No. 25, 29), tended to reduce total set, but afterchrome dyeing using chromable dyes tended to increase total set (No. 32–37). Neutral to alkaline methods of dyeing, with or without chrome, gave maximum total set approximately equal to that given by afterchrome dyeing in moderately acid dye liquors.

(*d*) *Permanent Set and Supercontraction in Absence of Chrome*

For simplicity the results are first considered of treatments (detailed in Tables I–III) in acid to alkaline solutions which are not complicated by the addition of oxidising or reducing agents. These show that the trends in permanent set (i.e., set remaining after release of the fibre in boiling water for 60 min) in relation to the pH of treatment are more in approximate agreement with the results of Blackburn and Lindley [7] than with those of Speakman [17]. Generally, slightly alkaline solutions produced a high degree of set. At lower pH the amount of set decreased and a moderate degree of supercontraction was found after the most acid treatments. With the exception of mordant dyes applied and chromed at ca. pH 3·5, the dyes, in amounts used commercially, had negligible influence on setting. Results similar in many respects were obtained by Okajima and Kikuchi [12] by studying the setting properties of dyed wool.

Treatment with formaldehyde (No. 7–9), which acts as a cross-linking agent and so delays the decay of tension by stabilising the fibre, produced supercontraction, the extent increasing with concentration of formaldehyde. On the other hand, treatments with reducing types of stripping agents (No. 10, 11), acting as a means of releasing tension by breaking disulphide bonds, produced a high degree of permanent set. These treatments are interesting for their relevance to the use of bisulphite as a setting agent [11]. The production of a high degree of permanent set in stripping can, however, be avoided by the use of methods (e.g. No. 12) which do not involve attack on the disulphide bond.

The possibility of cross-linking [19] occurring in dyeing with Procion Yellow RS (C.I. Reactive Yellow 4) was examined in No. 13b (Table I). The differences in set found by comparing the dyed fibres with the "blank"-treated fibres (No. 13a) were too small to indicate cross-linking with certainty under the conditions used.

With release in boiling water for 1 h as the criterion of permanence of set, comparison between the stability of set produced at the boil and that produced at higher temperatures (Table III) is complicated by (*a*) the tendency for set to resist release at any temperature below that of the original treatment, and (*b*) the tendency for tension to remain in the fibres because of the shorter treatment times of high-temperature dyeing. These

conflicting factors can account for the complexity of the results for permanent set in Table III. Although, in comparison with the corresponding results for treatments at the boil, all treatments at higher temperature gave enhanced total set, the degree of permanent set varied.

(e) *Permanent Set and Supercontraction in Presence of Chrome*

In considering the influence of prior chemical treatments on supercontraction, it is important to take into account the decay of tension [7] which will have occurred before the significant chemical reaction takes place. Decay of tension commences from the moment of stretching the fibre and is assisted by conditions which produce permanent set, though a considerable amount of tension will decay in acid liquors at 100°C. After tensioning, each fibre was held for about 30 min in water at room temperature before being introduced into the treatment liquor; thus, in every experiment, some of the initial tension will have decayed before the "dyeing" treatment began, but the subsequent rate of decay will have varied according to the conditions used in the remainder of the experiment. Treatment liquors near the boil will have caused relatively rapid decay of tension unless a stabilising or cross-linking agent was present. In afterchroming, an agent of this type was introduced half-way through the treatment process, i.e. when most of the tension had decayed. The result in most instances was to enhance the degree of permanent set which would have been obtained in the absence of chrome, but in two instances in which, because of the low pH (No. 24) or the short duration of treatment (No. 57), more residual tension was present during chroming, small amounts of supercontraction were obtained on release in boiling water.

Chrome-mordant methods with chromic acid (No. 30, 31, 40, 42, 44, 59) invariably produced supercontraction during release by boiling for 60 min in water, but the corresponding treatments under approximately neutral to alkaline conditions produced a considerable amount of permanent set owing to the influence of pH conditions known to produce set. Release for 15 min at the boil produced intermediate loss in set, which, because of the presence of residual stress, produced supercontraction in those chrome-mordant treatments with chromic acid which gave large amounts of supercontraction in the 60-min release. Chrome-mordant treatments with trivalent chromium in the form of chromium trifluoride (Table II, No. 26, 40) produced permanent set, whereas under similar conditions chromic acid produced supercontraction; thus the oxidising function of chromic acid in chrome mordanting contributes to the production of supercontraction or reduction in permanent set, probably by oxidation of keratin side-chains other than those which contain the disulphide bond [30].

A further examination of the relation of the valency state of chromium to its setting properties was carried out in a special series of treatments in which tensioned fibres were boiled at pH 3·4–3·5 in formic acid for increasing times between 10 and 90 min, (*a*) without chromium, (*b*) with chromium trifluoride, and (*c*) with potassium dichromate. The fibres were introduced into the liquor 5 min after stretching had ended, the ionic strength of the liquor was maintained approximately constant by adding sodium chloride, and the amount of chromium compound adjusted to give ca. 0·35% chromium in the treated fibres, as assessed from the analyses of the accompanying pieces of knitted fabric of similar fibre quality. The results of the release of the fibres in boiling water for 60 min are given in Fig. 1, which shows that the setting curve for the treatments with trivalent chromium indicates slightly higher set than that without chromium, whereas chroming with hexavalent chromium delays the development of set and so aids supercontraction. Further study appears necessary in order to establish the mechanism.

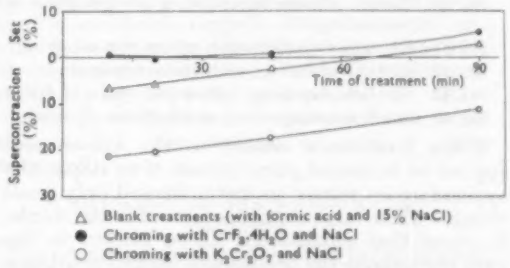

FIG. 1—Effect of chroming treatments (0·35% Cr) at pH 3·4–3·5 on setting

Dyeing on a chrome mordant tended to diminish the supercontraction obtained in the corresponding "chrome only" treatments with chromic acid, probably by frictional resistance created by the chrome lake interposed between the keratin chains.

The afterchrome treatments in acid solutions generally gave permanent set increasing with pH and chrome content and, in the treatments using stronger acids than acetic, a higher degree of set was obtained in the presence of chrome lake than with the reduced chrome mordant alone. The higher degree of permanent set is reflected in higher total set. Comparison of acid afterchrome treatments with the corresponding chrome-mordant treatments (Table II, No. 33, 30; 34, 31; 43, 42) invariably shows permanent set for the afterchrome and supercontraction for the chrome-mordant treatments, and the differences can be large.

ASSEMBLIES OF FIBRES

(a) *Effect of Steaming on Tufts of Fibres Dyed under Tension*

Material—The root two-thirds of 64s fleece wool staples was cleaned as described above for single fibres.

Method—Staples weighing 0·5 g were stretched quickly by 40% on a frame in water at room temperature, after which the broken fibres were removed and the intact fibres "dyed" by methods found to give extremes of permanent set and supercontraction on the basis of the results obtained with single fibres, i.e., as in No. 11, 20, 21, 22a, 25, 42, 43 (Tables I–II). The fibres were slackened during rinsing, steamed for 15 min, and dried at

TABLE IV

Set of Twist in Cross-bred Roving

(12s worsted count, 56–58s quality, 12 turns/in.)

No.	Treatment	Twist set (% of original) To water at 20°C for 20 min	Twist set (% of original) To air after subsequent steaming for 15 min	Cr as $K_2Cr_2O_7$ on dry wt. (%)
65	As No. 25 (Table II)	64	59	2·2
66	As No. 61 (Table III)	64	61	1·3
67	As No. 17 (Table I)	85	91	—
68	As No. 43 (Table II)	82	93	1·4
69	As No. 22a (Table I)	35	20	—

room temperature, and the tuft was mounted by one end on a card for inspection. Visual assessment of the tufts with regard to their appearance and length before steaming gave the following results—

No. 11 Little change in appearance
No. 20 Obvious shrinkage with return of some crimp
No. 21 Return of much crimp giving the *appearance* of shrinkage
No. 22a Obvious shrinkage with return of much crimp
No. 25 Obvious shrinkage with crimp retained
No. 42 Obvious shrinkage with small return of crimp
No. 43 Small shrinkage with small return of crimp

When treatments similar to the above were applied to tensioned yarn instead of to staple, the appearance on release in steam showed only small changes where large changes occurred in the staple. It seems that untensioned fibres, present in the yarn throughout the treatments, offered resistance by inter-fibre friction to the movement of tensioned fibres during the release in steam.

(*b*) *Setting of Twist in Yarn*

In order to examine the influence of dyeing on the tendency of yarn to retain twist, several treatments, selected for their ability to give extremes of permanent set and supercontraction in single fibres, were applied to 3½-in. lengths of 56–58s quality low-twist roving of 12s worsted count, after twisting to 12 turns/in. and clamping at both ends. Duplicate treatments were given to two samples of ether-extracted roving, each end of which was cemented to a short length of nichrome wire. About 30 min elapsed between the introduction of twist and immersion in the "dye" liquor. The amount of twist set by the treatment was found by difference from the original, after suspending the treated roving with the lower end free under a weight of 0·179 g and counting the number of revolutions made by the roving (*a*) whilst dry in air and (*b*) subsequently in water for 20 min at room temperature; (*c*) finally, the residual twist left after steaming the wet roving for 15 min and drying in air, was found by untwisting in a twist tester. (Release in boiling water proved unsuitable because of the felting produced.)

Excepting No. 69 (Table IV), which untwisted 2·4% in air, the "dyed" rovings showed no tendency to untwist in air. As was found by Bekku *et al.*[21], the release of the twisted bundle of fibres in water at room temperature proved a sensitive test for setting, since in two instances (No. 67, 68) which in similar treatments with single fibres were effective in producing a high degree of permanent set of extension, more release of twist was obtained by this method than by steaming for 15 min with subsequent measurement air-dry. It seems from No. 67 and 68 that the greater tendency to untwist in water than in steam is a result of the swelling action of water on tightly twisted roving which had received a high degree of permanent set. This behaviour contrasts with that of the other treatments (No. 65, 66, 69) in which the roving untwisted further in steam, apparently as a result of axial supercontraction of the fibres.

Under conditions of "dyeing" which would have led to supercontraction in length of single fibres, about 60% of the twist in the chrome-mordanted roving (No. 65, 66) was permanently set to release for 15 min in steam; and, as would be expected from conditions which produce a high degree of permanent set in tensioned fibres, No. 67 and 68 are instances of the permanent setting of more than 90% of the twist to release in steam for 15 min. Only No. 69, which represents a solvent-assisted method of dyeing at a low temperature, produced a relatively low degree of set in twist.

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Influence of Polar Groups on the Hydrophile–Lipophile Balance of Non-ionic Compounds

N. SCHÖNFELDT and U. ALMROTH

Compounds containing an oleyl radical and the same number of ethylene oxide units but different polar groups differ in their solubility in water and in their hydrophile–lipophile balance. A comparison was made by determining the cloud points in 10% sodium chloride solution and in dioxan–water, as well as by means of the water numbers. The oleic acid–polyglycol esters have the lowest solubility in water, because of the presence of diesters. The oleyl alcohol adducts are, at the same ethylene oxide chain length, probably three ethylene oxide units less hydrophilic than the oleylamine adducts. The solubilising power of a carboxyl group appears to be equal to that of a hydroxyl group and to that of three ethylene oxide groups. The hydrophilic character of an amine group is greater and is approximately equal to that of four ethylene oxide units.

Introduction*

The hydrophilic part of many non-ionic compounds contains one or more ethylene oxide units. Addition of ethylene oxide, or the introduction of a polyethylene glycol chain, generally occurs at a polar group attached to the hydrophobic material. Such polar groups contain one or more reactive hydrogen atoms; they act as a more or less stable link between the hydrophobic part of the compound and the polyethylene glycol chain.

We have been interested in the extent to which the polar groups influence the hydrophile–lipophile balance of the compounds, and have, therefore, determined this quantity for some non-ionic products containing, as the hydrophobic part, an oleyl radical, and as polar groups, –COOH, –OH, and $-NH_2$. It was obtained by determining the cloud point (*a*) in a 10% sodium chloride solution and (*b*) in a mixture of dioxan and water. In addition, the water number [1] was determined.

Different numbers of ethylene oxide units were added to oleyl alcohol and to oleylamine. Addition of ethylene oxide to oleic acid always leads to formation of diesters to a considerable extent, together with monoesters. In order to increase the percentage of monoesters in such a mixture excess of polyethylene glycol was used in esterification.

Experimental

STARTING MATERIALS

These had the following characteristics—

Oleic acid	
acid value	203
saponification value	203
iodine value	89
water content (%)	0·18
Oleyl alcohol	
hydroxyl value	211
iodine value	92
water content (%)	0·24
Oleylamine	
melting point (°C)	17·5
molecular weight	262
nitrogen (%)	4·93
secondary amine	0
iodine value	82·5
bromine value	58·6
water content (%)	0·13

* A brie preliminary notice of this paper was given in the book *Oberflächenaktive Anlagerungsprodukte des Äthylenoxyds, ihre Herstellung, Eigenschaften und Anwendung*, by N. Schönfeldt (for review see J.S.D.C., **77** (Mar 1961) 118).

PROCEDURE

Ethylene oxide adducts were prepared in the usual way in presence of an alkaline catalyst at high temperature and pressure. Polyethylene glycol mono-oleates (approx. 80% monoester content) were made by esterifying oleic acid with polyethylene glycol at a molar ratio of 1:3, sulphuric acid (0·5% by wt.) being used as catalyst. Polyethylene glycol dioleates were prepared at a molar ratio of 3:1, the amount of catalyst being unchanged.

PURIFICATION OF THE ESTERIFICATION PRODUCTS

The esters were purified according to the method of Matell [2] by shaking with benzene, ethyl alcohol, and a 10% sodium carbonate solution. The organic phase was then washed with a 50% ethyl alcohol solution, dried, and freed from solvent at 100°C *in vacuo*. This fraction was combined with one obtained by extracting the aqueous phase with benzene, drying the extract, and evaporating at 100°C *in vacuo* to remove solvent. Free polyglycols were then removed by the following procedure.

REMOVAL OF FREE POLYGLYCOLS

In accordance with the method of Weibull [3], samples were shaken with 5-N sodium chloride solution, ethyl acetate was added and the samples were again shaken. Two layers were formed, the lower one being the salt solution containing the polyethylene glycols, and the upper layer ethyl acetate containing the dissolved non-ionic compound. After repeated washings (sometimes up to six) of the ethyl acetate solution with 5-N sodium chloride solution the polyglycols were almost completely removed. After the solvent had been evaporated and any adhering sodium chloride separated, the purified compounds were ready for investigation.

ESTER CONTENT

Mono- and di-ester contents, and the number of ethylene oxide units in the polyethylene glycol oleates, were calculated as follows—

$$\frac{560x}{OH} = M_s + 44y$$

$$\frac{2 \times 560(100-x)}{F-OH} = 2M_s - 18 + 44y$$

where y = average number of ethylene oxide units in the mono- and di-esters (assumed to be equal),

x = monoester content (% by wt.), $100 - x$ = diester content (% by wt.), OH = hydroxyl value (mg KOH/g), M_s = molecular weight of fatty acid, and F = saponification value (mg KOH/g).

CLOUD POINT AND WATER NUMBER

Cloud points in 10% sodium chloride solution (CP_s) were determined by heating a 1% solution of the compound in the solvent until cloudiness appeared. Cloud points in a mixture of dioxan and water (CP_d) were measured by dissolving 0·2 g of the product in 20 ml of the mixed solvent (43:57 by wt.) and heating the solution until cloudiness appeared.

Water numbers were determined according to a slight modification of the titration method of Greenwald *et al.*[1]. Samples (3 g) were weighed in Erlenmeyer flasks and dissolved in 50 ml of a dioxan–benzene mixture (96:4 by wt). The solution was titrated with distilled water at 21°C until the first persistent turbidity appeared. The water number is the volume (ml) required to produce turbidity; the end-point was generally sharp. Freshly prepared stocks of solvent mixture were carefully analysed before use.

NON-IONIC COMPOUNDS

Tables I–III list the characteristics of the products used.

Results and Discussion

(*a*) CLOUD POINTS IN 10% SODIUM CHLORIDE SOLUTION (CP_s)

The cloud points of oleyl alcohol– and oleylamine–ethylene oxide adducts of different ethylene oxide chain lengths are shown in Fig. 1.

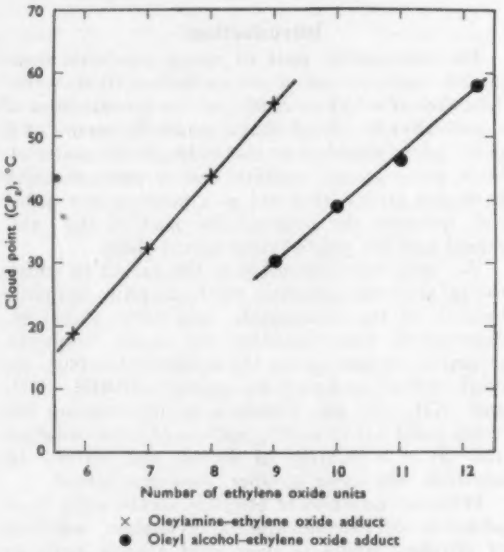

FIG. 1— Cloud points in a 10% sodium chloride solution of some oleyl alcohol– and oleylamine–ethylene oxide adducts

The difference between the number of ethylene oxide units in the oleylamine adduct and the number in the oleyl alcohol adduct at three cloud points (32, 43·5, 55°C) is approximately 2·3, 2·6, and 2·9, respectively. The third value is probably the most reliable one, since it is obtained with the compounds containing the longest ethylene oxide

TABLE I

Characteristics of Oleyl Alcohol–Ethylene Oxide Adducts

Average number of ethylene oxide units calc. from the OH value	Iodine value	OH value	Iodine value
0	0	211	92
3·8	3·5	129·4	59·2
4·8	4·5	118·0	53·5
5·9	5·7	106·9	48·3
6·5	6·4	101·8	45·6
7·3	7·2	95·3	42·9

TABLE II

Bromine Values of Oleylamine–Ethylene Oxide Adducts

Average number of ethylene oxide units calc. from the bromine value	Bromine value
0	58·6
2·0	44·6
3·2	38·8
4·0	35·8
5·1	32·2
5·7	30·6

TABLE III

Characteristics of Polyglycol Mono- and Di-oleates

Average number of ethylene oxide units	Acid value	Saponification value	OH value (mg KOH/g)	Monoester (%)	Diester (%)
0	203	203	—	—	—
3·9	0·1	127·4	99·7	80	20
3·9	0·5	158·7	1·3	1	99
5·2	—	—	88·7	80	20
5·2	—	—	0	—	100
6·5	0·6	103·6	81·6	81	19
6·5	1·2	135·9	0	—	100
7·4	0·5	96·8	74·7	80	20
7·4	1·3	129·6	0	—	100
8·7	1·0	125·3	0	—	100
10·7	0·9	—	63·5	83	17
14·0*	—	—	—	78–80	20–22
18·0*	—	—	—	78–80	20–22
21·1	1·6	—	38·0	78	22

* Made by mixing polyglycol oleates with 10·7 and 21·1 ethylene oxide units, respectively.

chains. These products contain less unchanged starting material which could influence the hydrophile–lipophile balance.

(*b*) CLOUD POINTS IN DIOXAN–WATER MIXTURE (CP_d)

The hydrophile–lipophile balance of ethylene oxide compounds can be measured by the cloud point in a 10% sodium chloride solution only if they are markedly hydrophilic. To study ethylene oxide adducts with comparatively short ethylene oxide chains, which are not completely soluble in water, cloud points were determined of their solutions in a mixture of dioxan and water. The results are plotted in Fig. 2.

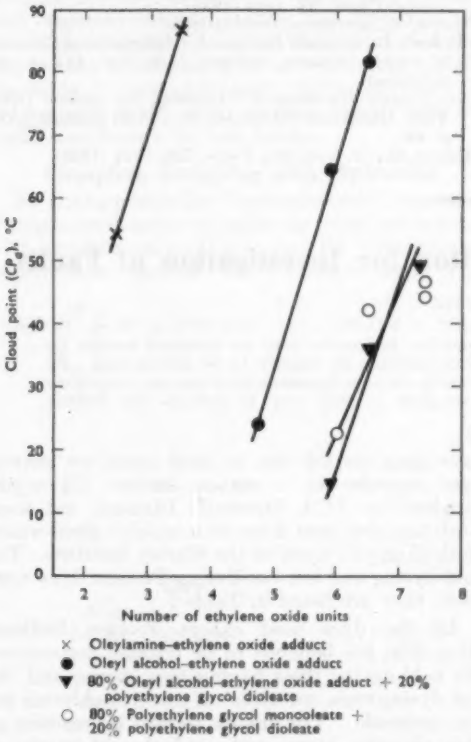
Cloud point (CP_d), °C
Number of ethylene oxide units
× Oleylamine–ethylene oxide adduct
● Oleyl alcohol–ethylene oxide adduct
▼ 80% Oleyl alcohol–ethylene oxide adduct + 20% polyethylene glycol dioleate
○ 80% Polyethylene glycol monooleate + 20% polyethylene glycol dioleate

FIG. 2— Cloud points in a dioxan–water mixture of some non-ionic compounds

The oleylamine–ethylene oxide adducts contain, at two representative cloud points (54 and 81°C), about three ethylene oxide units fewer than the corresponding oleyl alcohol adducts. Fig. 2 also contains a plot of the cloud points of polyglycol oleates containing 80% monoester and 20% diester dissolved in dioxan–water. Because of the presence of diesters this curve cannot be directly compared with those of the other adducts, since the diesters affect the cloud points. To confirm these results we added 20% of polyglycol dioleates containing an ethylene oxide chain of the appropriate length to oleyl alcohol–ethylene oxide adducts. The curve obtained was similar to that of polyglycol oleates containing 80% monoester and 20% diester.

(*c*) WATER NUMBERS IN DIOXAN–BENZENE

The hydrophile–lipophile balance of these compounds has also been estimated by the water number method. The results obtained are plotted in Fig. 3.

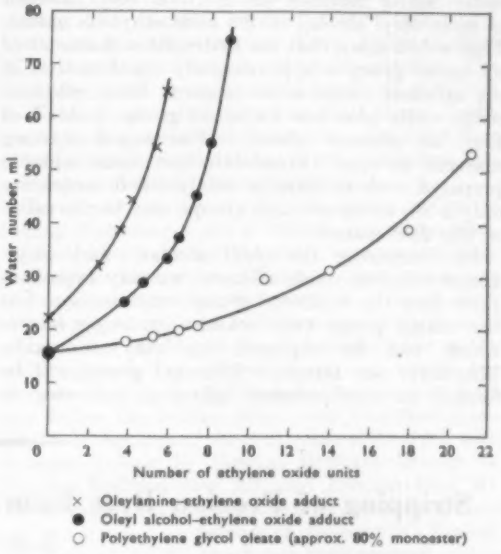
Water number, ml
Number of ethylene oxide units
× Oleylamine–ethylene oxide adduct
● Oleyl alcohol–ethylene oxide adduct
○ Polyethylene glycol oleate (approx. 80% monoester)

FIG. 3— Water numbers of some non-ionic compounds

The oleylamine–ethylene oxide adducts contain, at equal water numbers, approximately three ethylene oxide units fewer than the oleyl alcohol adducts. The water numbers increase slowly with ethylene oxide chain length at first, probably because of the presence of unchanged starting material, but then more steeply.

The water numbers of polyglycol oleates containing 80% monoester and 20% diester are lower than those of the other two types of ethylene oxide adduct.

The water numbers of oleic acid and oleyl alcohol are equal (15·3), but that of oleylamine is higher (22·2). A difference in the water numbers may be attributed to the solubilising power of the polar groups. Comparison between low-molecular-weight straight-chain aliphatic acids, alcohols, and primary amines indicates that butyric acid (but not valeric acid), propyl alcohol (but not butyl alcohol), and butylamine (but not amylamine) are soluble in all proportions in water [4]. Consequently, a carboxyl or a hydroxyl group has the power to solubilise three methylene (methyl) groups. However, a primary amine group solubilises four. Thus, there is some correlation between the solubilising power of these polar groups and their water numbers.

According to Cohen [5], a hydroxyl group can solubilise three methylene groups. Consequently, each ethylene oxide unit can solubilise one methylene group, because ethylene oxide in itself contains two methylene groups. The solubilising power of a hydroxyl group is therefore equal to that of three ethylene oxide units. If a carboxyl group is as hydrophilic as a hydroxyl group, its solubilising

power will also correspond to that of three ethylene oxide units.

The hydrophilic character of an amine group is probably equal to that of about four ethylene oxide units. From Fig. 3, oleylamine is seen to have the same water number as 2·9 etho-oleyl alcohol (1 mole oleyl alcohol + 2·9 mole ethylene oxide). This would mean that the hydrophilic character of an amine group is approximately equal to that of six ethylene oxide units (approx. three ethylene oxide units plus one hydroxyl group) instead of four, as claimed above. Unchanged starting material in oleyl alcohol–ethylene oxide adducts prepared with an alkaline catalyst and containing only a few ethylene oxide groups may be the cause of this discrepancy.

In comparing the oleyl alcohol– and oleylamine–ethylene oxide adducts, we may even consider that the hydroxyl group contains one, but the amine group two, reactive hydrogen atoms which can be replaced by ethylene oxide. Therefore, one terminal hydroxyl group will be formed in oleyl alcohol adducts, but two in oleylamine adducts. As the solubilising power of a hydroxyl group is assumed to be equal to that of three ethylene oxide units, the hydrophilic character of oleylamine adducts containing two terminal hydroxyl groups will be, at equal ethylene oxide chain lengths, three ethylene oxide units higher than that of the oleyl alcohol adducts. The results described seem in general to confirm this conclusion.

BEROL AB
GOTHENBURG
SWEDEN

(*Received 27th October 1960*)

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Stripping of Procion Dyes from Cotton for Investigation of Faults

T. P. NEVELL

Procion-dyed cotton that is insoluble in cuprammonium hydroxide may be rendered soluble by treating it in boiling 4% sodium carbonate solution, thus enabling its fluidity to be determined. By means of hot 40% aqueous hydrazine, all except one of twenty Procion dyes examined may be completely stripped from cotton without degrading it. Cloth stripped in this way is suitable for fluidity determinations and for re-dyeing for testing purposes.

General

One of the chief virtues of reactive dyes, e.g. Procion dyes, is that they combine chemically with the fibre being dyed. However, if it becomes necessary to remove the dye from the fibre, this virtue becomes a vice because a covalent bond has to be broken.

Apart from the rectification of mistakes in full-scale dyeing, which will not be discussed here, the removal of Procion dyes from cotton goods may be required when faults are being investigated. For example, it is frequently necessary to determine the fluidity of the material. This is impossible with many Procion-dyed materials (particularly those dyed with the cold-dyeing dyes) because they are insoluble in cuprammonium hydroxide solution and other solvents for cellulose. It is also sometimes necessary to strip fabric completely so that it can be re-dyed under carefully controlled conditions. Existing methods [1] for stripping Procion dyes are inadequate for either of these purposes. The method employing hot acetic or formic acid is liable to affect the fluidity that is to be measured, and stripping with alkaline hydrosulphite merely destroys the azo linkage of the dye without affecting the linkage between the triazine ring and cellulose. The experiments described below show how it is possible either to break sufficient of the linkages between dye and cellulose to enable fluidities to be measured, or to remove the dye completely, without degrading the cellulose. They have been carried out, in most cases, on heavily dyed samples of a cotton limbric ($3\frac{1}{4}$ oz/yd^2) supplied by I.C.I. Dyestuffs Division, but some work has also been done on a heavier plain-weave cloth (5 oz/yd^2) dyed at the Shirley Institute. Ten cold-dyeing and ten hot-dyeing Procion dyes were used; they are listed in Table I.

All the dyes used except Procion Brilliant Blue H7G are believed to be triazine derivatives, the cold-dyeing dyes having two atoms, and the hot-dyeing ones one atom, of reactive chlorine per dye molecule. The linkage formed by reaction of this chlorine atom with a hydroxyl group in cellulose would be expected to have properties intermediate between those of an ester and those of an ether linkage. The problem, therefore, was to find a reagent that would attack this linkage without degrading the cellulose. The most obvious type was hot aqueous alkali [2], and it was soon found that boiling with aqueous sodium carbonate hydrolysed sufficient cross-linkages in materials originally insoluble in cuprammonium hydroxide to render them soluble. Little dye was removed from the cloth, however, and another reagent was therefore sought for use when complete stripping is required. Hot aqueous hydrazine was found to be extremely effective; all the dyes except Procion Brilliant Blue H7G were completely stripped by it, and the cloth could be re-dyed perfectly evenly with the same dye. Details of these two methods of treatment follow.

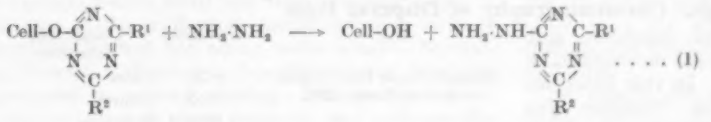
Partial Hydrolysis with Sodium Carbonate

Preliminary tests showed that all the dyeings that were originally insoluble in cuprammonium hydroxide could be made soluble by boiling them under reflux with 4% aqueous sodium carbonate for two hours. The sodium carbonate solution became coloured, but the depth of colour on the cloth was only slightly diminished. Table I shows that, although the fluidities of the cuprammonium-soluble samples (mostly those dyed with hot-dyeing dyes) were increased slightly by the treatment, they were not increased sufficiently to detract from their significance in testing. The increases were probably due to the presence of air during the soda boil. After treatment in boiling sodium carbonate solution, both originally insoluble and originally soluble samples had approximately the same fluidities. It is therefore concluded that the fluidity of Procion-dyed cotton that is initially insoluble in cuprammonium hydroxide may be determined if the cloth is first boiled with 4% sodium carbonate for two hours.

Complete Stripping with Hydrazine

By analogy with the "hydrazinolysis" of esters[3], Procion-dyed cellulose might be expected to react with hydrazine according to eqn. (1). Preliminary experiments showed that Procion dyes were rapidly and extensively removed from the dyed cotton samples by hot aqueous hydrazine. A range of concentrations from 4% to 64% was tried, and it was found that a 40% solution was the most effective. All the dyes in Table I, except Brilliant Blue H7G, were completely stripped by boiling the samples under reflux with 40% hydrazine for three hours. Even Brilliant Blue H7G was stripped to a considerable extent. The fluidities of the treated materials, which are given in Table I, are slightly lower than those of the corresponding soda-boiled materials and show that no significant degradation of the cellulose has accompanied the hydrazine treatment.

Treatment of pieces of cloth under reflux in boiling hydrazine is not a suitable procedure when it is intended to re-dye the cloth, because stripping is likely to be uneven. When re-dyeing tests are to be done, it is essential that the piece of cloth should not be allowed to become severely creased or to collect bubbles on its under-side while in the stripping solution. Experiments in which the temperature of the hydrazine solution was kept just below the boiling point were therefore made with 8-in. squares of the 5-oz plain cloth dyed with Procion Yellow R, Procion Brilliant Orange G, Procion Brilliant Red 2B, and Procion Blue 3G. The squares of cloth were treated for two separate periods of one hour in a beaker, which was kept covered with a watch-glass except when the cloth was being stirred with a glass rod. The operation was conducted in a fume cupboard



. . . . (1)

TABLE I

Procion dye	*Colour Index* No. C.I. Reactive	Fluidity of cloth in cuprammonium hydroxide (0·5% solution at 20°C) Original	After boiling with 4% Na_2CO_3 for 2 h	After boiling with 40% N_2H_4 for 3 h
None*		2·8	4·1	3·1
Cold-dyeing				
Brilliant Yellow 6G	Yellow 1	I‡	4·5	4·1
Yellow R	Yellow 4	I	5·1	4·0
Brilliant Orange G	Orange 1	I	5·0	4·6
Scarlet G	Red 8	I	5·0	4·4
Red G	Red 5	I	4·6	4·2
Brilliant Red 2B	Red 1	2·1†	5·0	4·4
Brilliant Red 5B	Red 2	3·8	4·7	3·9
Rubine B	Red 6	3·1†	5·0	4·5
Blue 3G	Blue 1	I	6·3	4·5
Brilliant Blue R	Blue 4	I	4·9	4·8
Hot-dyeing				
Brilliant Yellow H5G	Yellow 2	I	4·6	4·2
Yellow HA	Yellow 3	4·2	4·6	4·3
Brilliant Orange HGR	Orange 2	3·7	5·3	4·5
Scarlet H3G	Red 9	4·7	5·8	4·5
Brilliant Red H3B	Red 3	4·2	5·6	4·5
Brilliant Red H7B	Red 4	4·5	6·1	5·3
Rubine H2B	Red 7	5·1	6·3	5·4
Blue HB	Blue 2	5·6	6·6	5·9
Brilliant Blue H7G	Blue 3	4·5	4·8	4·8
Black HG	Black 1	I	6·1	4·6

* Original limbric.

† Gelatinous solution, suggesting incomplete dissolution.

‡ I = Insoluble.

because of the toxicity of hydrazine vapour. In each case a practically white cloth was obtained which could be re-dyed perfectly evenly with the same dye.

It has been reported that, when the colour of cotton dyed with certain Procion dyes is partially destroyed by means of alkaline hydrosulphite, the presence on the cotton of an organic molecule containing an amino group can be demonstrated by the deep colour developed when the reduced dyeing is diazotised and coupled with β-naphthol [4, 5]. This test has been applied to the twenty dyeings given in Table I and found to give a positive result with Brilliant Yellow 6G, Yellow R, Blue 3G, Brilliant Blue R, Brilliant Yellow H5G, Brilliant Orange HG, Brilliant Red H7B, and Blue HB. With the other twelve dyes no striking colour change occurred when the diazotised samples were coupled with β-naphthol. However, none of the twenty dyeings that had been stripped with hydrazine developed any appreciable colour on diazotisation and treatment with an alkaline solution of β-naphthol. This strongly supports the view that the linkage between the triazine ring and the cellulose is broken during stripping with hydrazine.

* * *

Some of the experimental work was done by Miss V. A. Morris and Miss D. M. Wynne.

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(*Received 28th November 1960*)

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[6] Present address: Department of Textile Chemistry, The Manchester College of Science and Technology, Manchester 1.

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Paper Chromatography of Disperse Dyes

For the paper-chromatographic separation and identification of disperse dyes some methods have already been recommended [1–8]. In this letter we describe our results in this field. Disperse dyes are azo or anthraquinonoid derivatives insoluble in water, but soluble in organic solvents. In an earlier paper [9] we have shown that such compounds must be chromatographed in solvent systems in which non-aqueous solvents are used as the stationary phase. Recently we have indicated that for separation and identification of simple aminoanthraquinone derivatives the solvent system 1-bromonaphthalene/pyridine:water (2:1 or 1:1) is suitable. We have now found that this solvent system gives excellent results with disperse dyes. We have chromatographed hundreds of disperse dyes and obtained in all cases good resolution and well-formed, round spots, the results being far better than those given by previously described chromatographic methods.

The following results are typical. They were obtained on Whatman No. 3 paper impregnated with a 1:10 (vol./vol.) solution of 1-bromonaphthalene in chloroform; the mobile phase was a 1:1 mixture of pyridine and water, saturated with 1-bromonaphthalene.

Dye	R_f values	Colour and Intensity
Celliton Fast Black BTN	0·04	brown
	0·16	violet
	0·29	orange
	0·33	greenish blue
	0·40	violet (faint spot)
	0·58	blue
	0·69	blue
	0·78	violet
	0·89	blue (faint spot)
Setacyl Black BSN Supra = Artisil Black BSN	0·35	violet
	0·53	yellow
	0·89	blue
Perliton Brown GR	0·04	red
	0·28	red
	0·51	yellow
	0·70	greyish blue
	0·83	greyish blue
Setacyl Brown RN Supra	0·23	red
	0·32	brown
	0·45	violet
	0·85	blue
Serisol Hosiery Brown GN	0·10	blue (faint spot)
	0·23	violet
	0·51	yellow
	0·60	violet
	0·87	blue

The following yellow disperse dyes have been found to be identical: Artisil Yellow 2GN* (two additional yellow spots), Cibacet Yellow 2GC*, Cibacet Yellow GGR, Serisol Fast Yellow GD*, Setacyl Yellow 2GN*, Setacyl Yellow 2GN Supra, Celliton Fast Yellow G*, Celliton Fast Yellow GR (one additional red spot), Celliton Fast Yellow GGR, Delfacet Fast Yellow GSD, Delfacet Yellow DG, Palanil Yellow G, Palanil Yellow GR (one additional orange-red spot) and Novalon Yellow 2GN*.

In some cases the following solvent systems have also been found of value—

1-bromonaphthalene/90% acetic acid
lauryl alcohol/ethanol:ammonia (1:1)
lauryl alcohol/ethanol:N HCl (1:1)

* Dyes listed in *Colour Index* as C.I. Disperse Yellow 3.

To obtain information quickly, it is often possible to use papers impregnated with formamide; hexane, benzene, chloroform, or mixtures of these compounds can be used as mobile phases.

Further details will be published shortly.

J. Gasparič
I. Táborská

Research Institute for Organic Syntheses
Pardubice-Rybitví
Czechoslovakia

25th January 1961

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[2] Zahn, H., *Textil Praxis*, **6**, 127 (1951); *Teintex*, **17**, 193 (1952).
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[5] Janoušek, J., *Faserforsch. und Textiltech.*, **8**, 339 (1957); J.S.D.C., **73**, 328 (1957).
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The Perkin Family

In the book *Perkin Centenary, London* and in other publications it is stated that Sir William Perkin's forebears came from Black Thornton, near Ingleton, Yorkshire [1,2]. I lived within three miles of Ingleton until I was nineteen, and knew and frequently visited Thornton-in-Lonsdale. It was never known as Black Thornton, but the neighbouring village of Burton-in-Lonsdale was known locally as Black Burton.

I have been in communication with Canon B. W. Simpson, Vicar of Thornton-in-Lonsdale, and I am indebted to him for the following information. He says—

"I know of no such place as Black Thornton, but Burton-in-Lonsdale, which was in Thornton-in-Lonsdale parish until less than a century ago, is known as Black Burton, partly because there is another Burton not many miles away— Burton-in-Kendal. Burton-in-Lonsdale was largely occupied in pottery making. At one time there were as many as seven potteries, and perhaps this caused much smoke. Also there was a little coal mining not far away. The Perkin entries on our registers are all described as 'of Burton'. I have no doubt that Black Thornton is an error for Black Burton in the parish of Thornton-in-Lonsdale.

We have no records in this parish of the Perkin family, except entries in the registers of some births and deaths. None of the persons mentioned in your letter are recorded. Two children of a Thomas Perkin (a *master shoemaker*) were baptised in 1816 and 1818 or thereabouts. There are other entries of the same surname."

In passing it may be noted that the book *Perkin Centenary, London* [1] contains the following paragraph—

"The link between the Perkin family and the Leathersellers' Company was an old one, established by Sir William Perkin's grandfather, Thomas Perkin, an apprentice to the *leather trade* in the City of London, who was 'made free by servitude' in 1790. Of a roving disposition, this ancestor, whose forebears were north-country yeomen, settled at Black Thornton, near Ingleton, in Yorkshire."

It seems probable, from the dates given, that the Thornton registers refer to a Thomas Perkin other than Sir William's grandfather. Whether there is any relationship between these two men is unknown.

My thanks are due to Canon Simpson for the trouble he has taken in searching the registers.

H. Wilkinson

44 Fulwood Hall Lane
Fulwood
Preston, Lancs

9th February 1961

[1] *Perkin Centenary, London* (London: Pergamon Press 1957), p. 26.
[2] Waters, H., *Manchester Guardian*, 7th May 1956.

ERRATA

Change in Colour of Vat Dyes on Soaping. J. Wegmann (J.S.D.C., **76** (Dec 1960) 679)— The first sentence of this letter should read "The abstract of the paper by Golomb [5] containing the criticism referred to above mentions only the title: 'Progress in Application of Vat Dyes in Dyeing and Printing'." In the third sentence the superscript numbers 3 and 5 should be transposed.

C.I. 61135 as Bluing Agent in Laundry Detergents. *BP* 850,531 (J.S.D.C., **77** (Jan 1961) 36)— This abstract should be deleted. It has been replaced by the abstract "Polar Brilliant Blue GAW (see C.I. Acid Blue 127) as Bluing Agent in Laundry Detergents", which appears on p. 179 of this issue, where there also appears an abstractor's note giving the reason for this change.

Salt-like Dyes for Acrylic Fibre Blends. The abstract with this heading (J.S.D.C., **77** (Feb 1961) 78) refers to the patent *BP* 853,985, and not to *BP* 853,955, as printed.

Notes

Midlands Section—Leicester Junior Branch

The Inaugural Meeting of the Leicester Junior Branch of the Midlands Section of the Society was held at the College of Technology and Commerce, Leicester, on 21st February 1961. The President of the Society, Mr. F. M. Stevenson, in inaugurating the Branch, emphasised that the future success of the Society depended on the goodwill and participation of younger members and on the interchange of ideas at all levels. The importance of the Society to Junior Members was not only to give them technical and scientific information through lectures and the *Journal*, but also to let them acquire a way of thinking and a familiarity with the background of the industry which would be a great help to them in their future careers.

The Principal of the College, Mr. R. E. Wood, welcomed the new Junior Branch, stressing the importance of technical and professional societies both to industry and to the student. Mr. W. A. Edwards, Chairman of the College of Technology Dyeing and Finishing Advisory Committee, recalled the formation of the Midlands Section itself some forty years before; it was gratifying, he said, to know that enthusiastic younger people were continuing the traditions laid down so long ago.

Officers and a Committee were elected to serve until the first Annual General Meeting to be held during April.

West Riding Section Competition for the Dyers and Finishers Association Prizes

The following prizes have been awarded as a result of the recent essay competition for Junior Members—

First Prize

Terence Michael Baldwinson

Second Prize (three prizes of equal value)

John Elliott
John Keith Hanson
Malcolm Britton Roberts

Mr. Baldwinson, who is 21, is a second-year part-time student at Bradford Technical College. He is employed as a laboratory assistant and has been in the industry for four years. The winning essay was entitled "The evolution of the disperse dyes and their application to the hydrophobic fibres". Publications Committee has decided to publish it in the *Journal* in the near future.

The prizes were presented by Mr. G. Darnton Wade, President of the Dyers and Finishers Association, after the West Riding Section A.G.M. on 23rd March 1961.

Merger of Research Associations

The merger of the British Cotton Industry Research Association and the British Rayon Research Association became effective on 1st April 1961. The new association, which is called the Cotton, Silk and Man-made Fibres Research Association, is housed at the Shirley Institute, Didsbury, Manchester.

Internationale Farbtagung 1961

The 1961 "Internationale Farbtagung" will be held in Düsseldorf on 23rd–26th May on the theme "Practical Applications of Colorimetric Knowledge". The topics to be covered will be colorimetry; physiology, psychology and aesthetics of colour; technical aspects of colour, e.g. dyeing; instruction and documentation. Full details are available from the Geschäftsstelle des Fachnormenausschusses Farbe (FNF), Berlin-Dahlen, Unter den Eichen 87.

11th Annual Technical Conference of the Hungarian Technical and Scientific Society of the Textile Industry

This Conference will take place in Budapest on 1st–3rd June 1961. Subjects of papers of interest to colourists include dyeing of synthetic fibres and finishing worsted fabrics containing synthetic fibres, use of reactive dyes, new finishing machinery, antistatic finishing, a comparative study of thickeners for printing, non-felt finishing of knitted wool fabrics, a coloristic study of Hanil Red BL Base, test methods for auxiliary products, Czechoslovakian and Soviet dyes, and material testing and quality control.

The papers will be given in Hungarian, but abstracts in German and possibly in English will be provided and, as far as possible, interpreters will be available.

An excursion is planned for 4th June, and on 5th and 6th June delegates will be able to visit various textile mills and institutions. Application forms are available from the Secretary of the Society, Budapest V, Szabadság Tér. 17, Hungary.

Laboratory Apparatus and Materials Exhibition

The 1961 Exhibition will be held at the Royal Horticultural Society's New Hall, London, on 19th–22nd June. A special feature of this exhibition is a series of twelve lectures (three each day) on recent advances in laboratory techniques. Information is available from the organisers, UTP Exhibitions Ltd., 9 Gough Square, Fleet Street, London E.C.4.

International Plastics Exhibition and Convention

The 1961 Exhibition will be held in the Grand, National, and Empire Halls, Olympia, London, on 21st June–1st July. This exhibition is organised by *British Plastics* with the co-operation of the British Plastics Federation; tickets and full information can be obtained from Iliffe Exhibitions Ltd., Dorset House, Stamford Street, London S.E.1.

Gordon Research Conferences

The 1961 Gordon Research Conference on Textiles will be held on 10th–14th July at Colby Junior College, New London, New Hampshire, U.S.A. The following papers will be presented—

Effects of Fibre Structure on Dyeability with Disperse Dyes.

Applications of Modern Thermoanalytical Techniques to Studies of Reactions at Elevated Temperatures in Fibres, Fibre-forming Polymers, and Elastomers.

Ideas and Investigations on the Super-molecular Structure of Fibrous Materials.

Order and Mobility of Molecules in Fibres.

Problems of Heat Setting of Nylon in High-speed False-twist Crimping Processes.

Isotactic Polypropylene Fibres.

Chemical Bonding and Reaction Rates of Wrinkle-resistant Finishes with Cotton.

Microscopically Observed Phenomena of Fibre Damage in Cotton.

Wool Stabilisation Through Interfacial Polymerisation.

Full details are available from Mr. W. G. Parks, Director, Department of Chemistry, University of Rhode Island, Kingston, Rhode Island, U.S.A.

Tritium in Self-luminous Paints

A self-luminous paint, Lumichron, has been developed which depends upon tritium for exciting the phosphor. Compared with radium-containing paints it has the advantage of higher initial brightness, longer luminous half life and lower energy level—with a maximum β-energy of 18 keV, radiation from tritium is too weak to penetrate an ordinary sheet of paper and so presents no external hazard. The costs per unit of light are comparable with those of radium. Application is much the same as with radium paint. In Lumichron the tritium is chemically bound in the molecule of a transparent plastic. Minute particles of crystalline phosphor are coated with the plastic. The tritium, being in intimate contact with the phosphor particles, causes them to glow. *Chem. Eng. News*, **39** (1) (2 Jan 1961) 56–57. C.O.C.

Death of Members

We regret to report the death of Mr. J. Holt, Mr. E. A. Millard, and Mr. F. R. Twemlow.

Meetings of Council and Committees
March

Council— 1st
Finance and General Purposes— 14th
Examinations Board— 16th
Colour Index Editorial Board— 20th
Publications— 21st

OBITUARY NOTICES

C. J. T. Cronshaw

Cecil John Turrell Cronshaw, President of the Society from 1939 to 1946, died in Manchester on 5th January 1961. Born on 13th June 1889, Cronshaw was educated at Bury Grammar School and later at Manchester University, where he obtained a First Class Honours Degree in Chemistry. In 1915 he joined Levinstein Ltd. Thus as a young man of 26 he entered the British dyestuffs industry at a time when there was bitter controversy about its neglected state and its inadequacy to meet Britain's wartime needs.

James Baddiley, who had joined the firm in 1909, had patiently built up a small research team which became the springboard for wartime effort. Cronshaw was first appointed as Baddiley's special assistant. Widely different in temperament and personality, these two understood each other. In their different ways both became dedicated men. They worked together intimately for over thirty years— Baddiley as the architect of a sound research tradition and Cronshaw eventually as the leader guiding the British dyestuffs industry through its re-birth in a period of economic and political difficulty.

In 1916 Levinstein Ltd. had acquired the former Meister Lucius and Brüning indigo factory at Ellesmere Port, which had been built to comply with the required working of foreign inventions under the revised Patent Act of 1907. The factory had been designed to operate on imported German phenylglycine and this intermediate had not been manufactured in Britain. The wartime need for indigo was urgent. Cronshaw's first major job in the industry was to put this factory into operation, a task which he and his colleagues completed within three months.

After the Armistice, Cronshaw became for a year the Chemical Controller of the Rhineland Area Factories in Germany. There he met leading members of the German dyestuffs industry who later came to regard him as the new leader of a revivified British industry.

In 1919, British Dyes Ltd, Huddersfield, merged with Levinstein Ltd. to form British Dyestuffs

Corpn. Ltd. Cronshaw returned to England to become Assistant to Dr. Herbert Levinstein, the then Managing Director. It was a time of difficulty and confusion. There was Government representation on the Board of the Corporation but scarcely any consistent Government policy. The import of German reparation dyes, following the Sankey Judgment in December 1919, had nearly ruined the struggling British Industry, and the introduction of the Dyestuffs (Import Regulation) Act in 1921 was almost too late to save it. In 1921 Cronshaw became Works Manager of the Manchester (Blackley) Factory of British Dyestuffs Corpn. Here he was engaged in the manufacture of some of the earliest rubber chemicals made on a commercial scale in Britain.

In 1924 Dr. Cronshaw was appointed Technical Manager of the British Dyestuffs Corpn. It was still a time of political flux. Sir William Alexander resigned as Chairman and Managing Director in 1924, and Dr. E. F. Armstrong succeeded him briefly in 1925. Then, in 1926, came the formation of Imperial Chemical Industries Ltd, of which British Dyestuffs Corpn, because of its importance in the organic chemical field, was one of the original constituents. The Corporation was renamed I.C.I. Dyestuffs Group and Cronshaw became its first Technical Director. In 1931 he became Managing Director of the Group and finally its Chairman from 1939 until 1943, when he became a Director of the Main Board of Imperial Chemical Industries, serving on it until his retirement in 1952.

Thus for almost thirty years Cecil Cronshaw's great talents and restless energy were devoted to the guidance of what is now I.C.I. Dyestuffs Division. Throughout, and in good time or ill, he encouraged and supported the research of the unit. He was delighted when, in the late 1920's, inventions of major importance began to come from the research laboratories at Blackley. He believed that research should be exciting and vivid; as he put it— "There should be a catch in the breath". He was profoundly interested in organisation, but only if it facilitated interplay between mind and mind.

Like most busy men he still found time for a multitude of external interests and public work. He was a Governor of Bury Grammar School and a member of the Court of Governors of the Victoria University of Manchester and of its Council for many years. He guided our own Society as President through the difficult war years, following a period as Vice-President of the Society of Chemical Industry (1935–38). He was a Prime Warden of the Worshipful Company of Dyers (1949–50). In this and in his Presidency of the Society of Dyers and Colourists he took particular pride and pleasure.

Of small stature, he was fizzing with energy in his prime. Whilst he loved the broad humour of the North of England and could tell a dialect story effectively, he had a natural relish for satire. He himself had a lightning and sometimes astringent wit, but was always happy to take as good as he gave. Unless it was unavoidable he never missed the annual smoking concert in which his staff lampooned with good-humoured satire the foibles of the organisation he had created. He enjoyed the fun with gusto, but he was also shrewd enough to note where the "grouse" had an underlying seriousness and make adjustments accordingly. He was always readily approachable if a problem was troubling one of his juniors. Rarely in such circumstances, however, did he propound a ready-made solution or decision. He preferred to lead the younger man by question and illustrative parable to propound his own solution and to make his own decision. Thus he often got the best out of the eccentrics and originals in his organisation. Throughout his life he remained a chemist at heart, and the University of Leeds recognised this with the award of an Honorary D.Sc.

Cronshaw was a wide and voracious reader with an avidity for new experience. At the same time he was happy to snatch a few hours at the County Cricket Ground and he delighted in the conflict of character in a Lancashire *v.* Yorkshire match. He was also a frequent attender at the Hallé Concerts and had a great enthusiasm for sailing.

Unhappily, the years of his retirement, which should have given him the leisure and energy to indulge his wide-ranging interests, were marred by failing health. A happier thought is that the award of the Society's Perkin Medal a year ago gave him enormous pleasure. He was very proud to receive a medal which bears the name of the founder-father of the industry which he had served so long and with such splendid devotion.

C. Paine

Dick Carter

The death occurred on 3rd January 1961 of Dick Carter. Carter, who was 59, was a Yorkshireman. He graduated at Leeds University, afterwards obtaining the degree of M.Sc.

He had a distinguished career, becoming a Fellow of the Royal Institute of Chemistry, a Fellow of the Society of Dyers and Colourists, and a Fellow of the Royal Society of Arts. For several years he was the chief colourist in the dyestuffs division of I.C.I., and afterwards assistant managing director of Wolsey Ltd., Leicester.

He later became a textile consultant and acted in this capacity for Winterbotham, Strachan & Playne Ltd. until he succeeded the late Mr. Marcus Stewart as the managing director of Strachan & Co. Ltd., in April 1955, being appointed Chairman the following year. He was also appointed a member of the Board of the parent company, Winterbotham, Strachan & Playne Ltd.

He served on the British Colour Council board of management and the joint technical committee of the Colour Users Association.

He also served on many technical committees of the Society, and particularly those concerned with fastness testing, and was a member of the Technical Advisory Committee. He was one of the original Fellows of the Society. By raising the standard of technical service and organising an extensive

programme of applied research at Blackley, he provided subjects for many papers read before the Society. Throughout his career he encouraged members of his staff to give the fullest support to all the Society's activities.

He took part in the recent formation and development of the West of England Section and was its Vice-Chairman at the time of his death. He is survived by his wife and by a son and daughter.

E. V. GILES
J. A. POTTER

T. Minshall

Thomas Minshall died at his home on 8th January 1961; he was 54 years of age. He was a Lancashire man who received his practical training at the Bolton works of Tootal, Broadhurst, Lee Co. Ltd, where he gained valuable experience in the application of fast dyes to cotton yarn. In 1937 he came to Huddersfield as head dyer for Messrs. J. L. Brierley Ltd, being primarily engaged in dyeing sulphur dyes on cotton yarn. He later joined the technical service laboratories of Messrs. L. B. Holliday & Co. Ltd.

All this valuable trade experience Minshall brought to bear in his work as Assistant Lecturer in Dyeing at the Huddersfield College of Technology, from September 1949 onwards. His studies at the Manchester College of Technology led to the award of the A.M.C.T, and in 1951 he was elected an Associate of the Textile Institute.

Minshall was in charge of the dyehouse at the College, and took part in departmental research; he was joint author of a paper on colour vision [J.S.D.C., **75**, 585 (1959)]. He was a member of the Society for nearly 40 years and served for some time on the "Committee on the Dyeing Properties of Vat Dyes".

Tom Minshall was a conscientious teacher, well-liked by his students and colleagues, to whom his sudden death came as a great shock. He was a keen churchman and served for several years as churchwarden of the ancient church of Almondbury, near Huddersfield. He is survived by his wife and a daughter.

F. JORDINSON

New Books and Publications

Technique of Organic Chemistry
Volume I
Physical Methods of Organic Chemistry
Parts III and IV

Edited by A. Weissberger. Third completely revised and augmented edition. New York and London: Interscience Publishers. 1960. Part III— Pp. xii + 1799–2634 + 13. Price, 184s. 0d. Part IV— Pp. xii + 2635–3539 + 65. Price, 195s. 0d.

Parts III and IV of Volume I of the *Technique of Organic Chemistry* have now been revised, and have been brought into line with modern thought and enlarged to take account of recent developments. Typical of this work, they are thorough in recording the essential detail of the physical method described, well referenced to allow the expansion of any topic by the specialist, and pleasantly readable.

Part III has been expanded to include "The Kerr Effect" and "Determination of the Faraday Effect". Part IV contains accounts of the following subjects— "Nuclear Magnetic Resonance", "Paramagnetic Resonance Absorption", "Determination of Transference Numbers", and "Controlled Potential Electrolysis"—as additions to the original work.

A considerable proportion of these two parts of Volume I is concerned with spectroscopy in one form or another. The well-established methods of ultraviolet and infrared spectroscopy are covered in Part III and include accounts of instrumental procedures for chemical analysis. The chapter heading "Colorimetry and Photometric Analysis" is misleading, for it is in no way concerned with the measurement of colour. Nevertheless, the general analytical aspects should be of considerable interest to workers in the dye field. Although little direct reference is made to dye systems, the fundamental requirements of instrumentation for this type of work, together with an account of effects leading to errors, are given. The emphasis is on basic theory and experimental techniques, leaving the "Practical Applications of Spectroscopy" to Volume IX. It is disappointing to find, in a work of this magnitude, little reference to British-made instruments, of which there are several of the most advanced design.

The account of fluorescence and phosphorescence is brief, but probably contains sufficient detail to be useful to the applied chemist who is intent on gaining some background information to these phenomena, which are of importance in dye chemistry.

Spectrometers for use in the ultraviolet and infrared regions are now indispensable tools for research in organic chemistry. Since the last edition of this book new spectroscopic techniques in the radiofrequency region have grown in importance. Nuclear magnetic resonance, paramagnetic resonance, microwave, and direct quadrupole spectroscopy are examples. The necessary theory is given for an understanding of these techniques the experimental arrangements for which are described in Part IV. The emphasis is on recent developments, and on results which are more likely to be useful to organic chemists. High-resolution N.M.R. spectroscopy is treated most fully as it is undoubtedly the most important. It is inevitable, however, that a report of this type will rapidly become out of date.

The accounts of potentiometry, conductivity determinations, and polarography in Part IV are well worth reading. The application of such methods to dye chemistry is important from the analytical and the more fundamental points of view. The general uses of such methods are adequately described and should permit the application of any of the methods to a particular analytical problem. The chapter on polarography is designed to instruct the organic chemist sufficiently in the essentials of the method so that he may devise his own analytical procedure.

Some detail is given of electrophoresis, but with respect to the moving boundary method rather than to paper electrophoresis. Nevertheless, some interest may lie here for those working in the protein field.

Part III contains a short chapter on light scattering which is of obvious interest in the polymer field, but it lacks detail. Considerable detail and wealth of reference are given for polarimetry, optical rotatory dispersion, and streaming birefringence. The last is of importance in the study of proteins and polymers; advances indicate that streaming birefringence will provide useful data on deformable macromolecules.

The coverage given to a diversity of physical methods makes Volume I extremely useful to the chemist, and a good source of information for the student of chemistry. For the applied chemist in the dye field there is no doubt that the spectroscopic information in Part III is the most useful, and is certainly worth consideration.

E. COATES

Chemistry and Chemical Engineering

By A. J. Bune. Pp. viii + 104. London: *The Sunday Times*. 1960. Price, 3s 6d.

This is the fourth of a series of Career Books, each dealing with a particular profession or trade. This book assumes that a scholar with a liking for science has just taken the General Certificate of Education at "O" Level or Scottish Leaving Certificate with Lower Passes, with some success in scientific subjects, and is thinking perhaps for the first time of a career in chemistry or chemical engineering. It should therefore by definition be of value to a careers master, say in a grammar school.

There is undoubtedly a need for clear and well-informed literature that will enable the student both to form his own views about his future course of study and to determine what satisfactions there are likely to be in his chosen career. For the latter purpose he needs to be given some picture of the industry that will stimulate his imagination. Mr. Bune's aim is to provide this sort of background. In doing so he tries to cover more than can reasonably be compressed into a little over 100 pages. The seven chapters dealing with research, the manufacture of chemicals in general, and some individual chemical product groups are too loosely written and insufficiently selective to enable the student to weigh the alternative possibilities very easily. The chapter on research is devoted mainly to the government research organisations, the nationalised industries, and research associations, together with a not very clear account of the discovery of polythene in private industry. It is perhaps not surprising that the chapter on conditions of service is almost wholly dominated by the salary structure and other conditions of the Scientific Civil Service, a subject upon which there is available publication, but it should have been possible, e.g. with the aid of the Royal Institute of Chemistry, to give some clearer account of the prospects of chemists in industry. On the technical side the book assumes too much knowledge in the student reader, and technical jargon— e.g. detergent alkylate— is sometimes used unnecessarily. Experienced readers will be surprised to learn "that most plastics can be made into fibres" or "spun out into long narrow shreds". Such looseness of writing does not set a good example to the aspiring young scientists.

All in all, although the student will undoubtedly find things of interest in this book, it will not help him as much as it could have done.

C. PAINE

Der Rakeltiefdruck
(Rotary Photogravure)

By A. Strakaty. Pp. 103, numerous half-tone illustrations of machinery, and coloured inserts. 11th Edition. Mauthausen: A. Strakaty. 1961. Price, DM 8.00.

The tenth edition was recently reviewed (J.S.D.C., **76** (July 1960) 444), and an idea of the scope and contents of this interesting annual was then given. The general plan of the new edition remains practically unchanged, but the contents have been much modified and there is, for instance, an improved treatment of the principles of rotary photogravure. It is in the treatment of new developments and new devices, both for the preparation of the rollers and for printing, that the new edition is most useful. Besides complete machines and procedures, it deals with topics such as new sensitive materials, screens, temporary supports and transfers, etching, and so on.

The catalogue of new models of printing machines is an impressive tribute to the German engineering industry in this field. Most of the inserts are specimens of fine colour printing by printing firms. Included is a series of beautiful examples of the subtle improvements in colour rendering obtained in passing from three-colour to four-colour printing.

H. A. TURNER

B.S. 3266:1960 Determination of Conductivity, pH, Water-soluble Matter, Chloride and Sulphate in Aqueous Extracts of Textile Materials

Pp. 12. London: British Standards Institution. 1960. Price, 4s. 0d.

Many official specifications set limits for the above quantities. As instructions for the preparation of the extract and for the performance of the measurements differ from one specification to another, Technical Committee D of the Textile Institute has drawn up a specification which includes two standardised procedures for extracting

textile specimens with water. The standard is based on T.T.S. No. 52, published in *J. Textile Inst.*, 1958, **49**, P 724.

An aqueous extract is prepared, using low-conductivity water if conductivity is to be measured; otherwise, high-grade distilled water may be used. Separate portions of extract are used for the measurement of pH and conductivity, and for each of the determinations of water-soluble matter, chloride (gravimetrically or volumetrically) and sulphate (gravimetrically). The water-soluble matter, and chloride and sulphate contents are expressed as percentages of the weight of the conditioned material. Conductivity and pH of the extract are expressed in the conventional units. M.T.

New Books Received

Origin Rules, Certification and Segregation of Stocks. Pp. iii + 42. London: Association of British Chemical Manufacturers. 3s. 6d. (2s. 6d. to members of A.B.C.M.).

A Statistical Manual for Chemists. EDWARD L. BAUER. Pp. x + 156. New York and London: Academic Press Inc. and Academic Press Inc. (London) Ltd. 1960. 38s. 0d.

Titration in Non-aqueous Solvents. A. H. BECKETT and E. H. TINLEY. Pp. iv + 56. 3rd Edition. Poole, Dorset: British Drug Houses Ltd.

Determination of Conductivity, pH, Water-soluble Matter, Chloride and Sulphate in Aqueous Extracts of Textile Materials. BS.3266:1960. BRITISH STANDARDS INSTITUTION. Pp. 12. London: British Standards Institution. 1960. 4s. 0d.

Determination of Wool Fibre Medullation. BS.3209:1960. BRITISH STANDARDS INSTITUTION. Pp. 6. London: British Standards Institution. 1960. 3s. 0d.

Methods for the Analysis of Water-soluble Coal-tar Dyes Permitted for Use in Foods. BS.3210:1960. BRITISH STANDARDS INSTITUTION. Pp. 34. London: British Standards Institution. 1960. 7s. 6d.

Chemistry and Chemical Engineering. ALAN J. BUNE. Pp. 104 + 4 pp. half-tones. London: The Sunday Times. 1960. 3s. 6d.

Manual of Man-made Fibres. C. Z. CARROLL-PORCZYNSKI. Pp. 304. Guildford, Surrey: Astex Publishing Co. 29s. 0d.

Technology of Non-metallic Coating. A. YA. DRINBERG, E. S. GUREVICH, and A. V. TIKHOMIROV (Translated from Russian and edited by E. BISHOP). Pp. xvi + 531. Oxford: Pergamon Press Ltd. 1960. 80s. 0d.

Silicones. S. FORDHAM (Editor). Pp. xi + 252 + viii pp. half-tones. London: George Newnes Ltd. 1960. 36s. 0d.

Biological Degradation of Cellulose. J. A. GASCOIGNE and M. M. GASCOIGNE. Pp. vi + 264. London: Butterworth & Co. (Publishers) Ltd. 1960. 42s. 0d.

Die Chemie der Applikation von Komplexfarbstoffen. H. GERSTNER. Pp. 202. Berlin: Akademie-Verlag. 1959. DM 25.00.

Laboratory Handbook of Toxic Agents. C. H. GRAY (Editor-in-Chief). Pp. viii + 170. London: Royal Institute of Chemistry. 20s. 0d.

Walzengravur und Schablonenherstellung im Textildruck. H.-G. JAKOB and F. WEISSGERBER. Pp. viii + 338. Heidelberg: Melliand-Verlag. 1960. DM 38.00.

Forschungsberichte des Landes Nordrhein-Westfalen. No. 761. Untersuchungen zur Rationellen Durchfärbbarkeit von Bastfasergarnen. I. LAMBRINOU-GEURTEN. Pp. 53. Cologne and Opladen: Westdeutscher Verlag. 1959. DM 14.10.

The Textile Industry in Europe (Statistical Study— 1959; Trend in 1960). Pp. 119. Paris: Organisation for European Economic Co-operation. 1960.

Britain's Scientific and Technological Manpower. GEORGE LOUIS PAYNE. Pp. xiii + 466. Stanford, U.S.A. and London: Stanford University Press and Oxford University Press. 1960. 45s. 0d.

Development of Shade Standard and Tolerances for USAF Blue 85 Service Overcoat (WADC Tech. Report 59–397). FRANK J. RIZZO, CONSTANTINE J. MEGAS, JOHN T. WALWOOD, and ALDO M. CRUGNOLA. Pp. viii + 58. Ohio: Wright Air Development Center. 1960.

Development of Shade Standards and Tolerances for USAF Tan 505 (WADD Tech. Report 60–294). FRANK J. RIZZO, CONSTANTINE J. MEGAS, ALVIN O. RAMSLEY, WILLIAM B. BUCHNELL, and RUTH J. EVANS. Pp. vii + 55. Ohio: Wright Air Development Division. 1960.

Development of Shade Standards and Tolerances for Silver Tan 193 (WADD Tech. Report 60–151). FRANK J. RIZZO, CONSTANTINE J. MEGAS, ALVIN O. RAMSLEY, JOHN T. WALWOOD, and RUTH J. EVANS. Pp. x + 112. Ohio: Wright Air Development Division. 1960.

Der Rakeltiefdruck. ADALBERT STRAKATY. 11th edition. Pp. 103 + ix col. plates. Mauthausen an der Donau: Adalbert Strakaty. 1961. DM 8.00.

Review of Textile Progress— Vol. 11 (1959). Pp. x + 448. Textile Institute, Society of Dyers and Colourists, and Butterworths. 1960. 60s 0d. (45s. 0d. to members of T.I. and S.D.C.).

Technique of Organic Chemistry, Vol. I, Part 3— Physical Methods of Organic Chemistry. ARNOLD WEISSBERGER (Editor). 3rd edition. Pp. xii + 1799–2634 + I1–I13. New York and London: Interscience Publishers Inc. 1960. 184s. 0d.

Technique of Organic Chemistry, Vol. I, Part 4— Physical Methods of Organic Chemistry. ARNOLD WEISSBERGER (Editor). Pp. xii + 2635–3539 + I1–I65. New York and London: Interscience Publishers Inc. 1960. 195s. 0d.

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Alginate Industries Ltd.

MANUTEX IN TEXTILE PRINTING— This loose-leaf manual contains a general survey of the properties and uses of the various brands of alginates marketed under this trade name, and a wide range of typical recipes in which Manutex is used for printing direct, acid, metal-complex, disperse, reactive, vat, solubilised vat, and oxidation dyes, and also in printing pigments, flock and metal powders with resin binders. There is also a comprehensive collection of patterns from bulk prints illustrating the results that can be obtained in roller- and screen-printing with these dyes on such diverse materials as raised-loop Terylene, Courtelle, Tricel, nylon voile, cotton–viscose rayon towelling, and a vigoureux print of a reactive dye (Drimarene Black Z–BL) on wool slubbing.

Badische Anilin- und Soda-Fabrik A.G.

INDANTHREN DYES ON COTTON PIECE— This card contains dyeings in three depths on cotton cloth of 73 Indanthren vat dyes applied by pigment padding. Full details of methods of application, etc. are provided in a separate booklet, while the usual very extensive data on fastness characteristics and dyeing behaviour are provided alongside the individual dyeings. Thus fastness figures for light (daylight and Fade-Ometer) and weathering are quoted for 2/1, 1/1, 1/3, 1/6 and 1/12 standard depth.

DYED SHADES ON UNWEIGHTED SILK— This card contains dyeings in two depths on silk yarn of 139 dyes, including basic dyes, acid dyes (Basolans), 1 : 1 metal complexes (Palatine Fast), 2 : 1 metal complexes (Ortolans) direct, chrome, and anthraquinonoid and indigoid vat dyes. It is noted that the Neopalatin and Vialon Fast dyes may also be applied to silk, but it is considered that they do not offer any advantage over the Palatin Fast and Ortolan dyes as regards colour, fastness, or ease of application.

WHITE DISCHARGES ON DYED ACETATE— This card contains 35 discharge prints on secondary cellulose acetate cloth dyed with selected members of the Celliton, Celliton Discharge and Celliton Fast ranges of disperse dyes. It is noted that all white discharges of grounds dyed with some Celliton Discharge dyes become yellow on prolonged storage or exposure to light. Vigorous aftertreatment of the prints with Nekanil AC reduces, and in some cases eliminates, this trouble at the expense of some loss in depth. Since the risk of this fault developing increases with increase in depth of colour, care should be taken to avoid overloading the material with dye, particularly when using mixtures for dark colours.

WHITE DISCHARGES ON DYED SILK— This card contains 30 discharge prints on dyed unweighted silk grounds. The dyes used include acid (Basolan and Basolan Fast), 1 : 1 metal complex (Palatine Fast), 2 : 1 complex (Ortolan) and Neopalatin dyes.

DIRECT PRINTING ON PERLON— This card contains 101 prints on Perlon polyamide fibre material. The dyes used include disperse (Perliton), metal-complex (Vialon Fast, Palatine Fast, Neopalatin, and Ortolan) and acid (Basolan and Basolan Fast) dyes, by the use of which a complete range of colours of satisfactory fastness to light and wet treatments may be obtained.

CELLITON FAST AND CELLITON DYES: DIRECT PRINTING ON ACETATE— This card contains direct prints on secondary cellulose acetate cloth of 50 disperse dyes particularly recommended for screen- and roller-printing. A number are available as pastes, and the range includes new dyes, designated by the letters GF, exhibiting good stability to burnt-gas fumes.

INDANTHREN, ANTHRA AND BRILLIANT INDIGO DYES IN DIRECT PRINTING ON COTTON FABRICS— This card contains 52 prints on cotton cloth of vat dyes applied by the Rongalit C–Potash process. A separate, comprehensive 90 pp. manual gives a general survey of direct printing by this method, detailed recipes and fastness data. The relevant technical data, principal fastness characteristics, and styles for which the dye is particularly suitable are appended alongside each pattern.

INDANTHREN OLIVE GR— This vat dye gives olive greens on cellulosic fibres and is particularly suitable for rainwear, service cloths and similar materials, when maximum fastness to light and wet treatments is demanded. It may be applied to all forms of material, the coverage of dead cotton is very good, and almost solid dyeings are obtained on cotton–viscose rayon mixtures. It is also of interest on silk. Fastness figures on cotton include— light 7, washing *c* (95°C) 4–5, chlorine *b* 4–5, chlorite *b* 2–3.

Ciba Ltd.

CIBACRON SCARLET 4GP— This reactive dye is recommended for dyeing wool in presence of Neovadine AN or AL, with Disperser CC added for pale dyeings. It gives very bright scarlets and is particularly recommended, in combination with Cibacron Brilliant Yellow 3G, for yellowish scarlets. It is more soluble than Cibacron Scarlet 2G. Fastness figures include— light (daylight) 5, severe alkaline milling 3–4, washing B 3.

CIBALAN BLACK BRANDS AS A BASIS FOR MUTED SHADES ON WOOL— This card contains dyeings on wool of three Cibalan Blacks, BGL, 2GL, and 2BL, in 1·5% and 3% depth, and twelve mixture dyeings based on these plus Cibalan Green GL, Orange RL, Brilliant Yellow 3GL, Yellow FGL, Browns BL and VRL, and Benzyl Fast Green F3G to give dark greys, browns, and greens. The amount of black used should not be less than 1%; Cibalan Greys BL or 2GL should be used for paler colours.

ORAFIX TR— This is an anionic emulsion of the o/w type of a resin precondensate for use as a binder in flock, matt, and metal-powder printing, and matt discharge printing on practically all types of fibres and fibre mixtures.

CIBA REVIEW NO. 140— Under the general title "Fluorescence", this issue contains articles on "Luminescence in Relation to Organic Compounds", "Fluorescent Brightness", "Optical Brightness in Textile Finishing", and "Evaluation of Whitening Effect and Fastness Testing of Optically Brightened Textiles". Technical articles include a description of the use of the various brands of Uvitex in detergents and Uvitex PRS in the paper industry, and the production of two-colour effects on wool by a one-bath method which exploits the fibre selective dyeing behaviour of the Cibacron reactive dyes when applied in absence of cationic levelling agents.

Ciba Clayton Ltd.

CIBACET DYES: PRINTS ON CELLULOSE TRIACETATE— This card contains direct prints on cellulose triacetate cloth of 40 disperse dyes, machine printed using crystal gum 1 : 3 as thickener, and steamed for 1 h at atmospheric pressure.

CIBACET ORANGE RF— This disperse dye gives bright reddish oranges on secondary cellulose acetate, cellulose triacetate, polyamide, and polyester materials. *o*-Phenylphenol and diammonium hydrogen phosphate are recommended as dyebath additions when Terylene is being dyed at 100°C. Since even very small amounts of this carrier can impair the light fastness of dyeings, it must be completely removed. This can readily be done by treatment in hot air at 190°C for 1 min. Fastness figures on Terylene include— light 7, sublimation (60 s at 180°C) 5, washing (Mechanical A–60°C) 5.

KITON FAST BLUE CRB— This level-dyeing acid dye gives bright reddish blues on wool and is recommended for dyeing yarn, particularly carpet yarn, and woven and knitted cloth. Cellulosic fibres are unstained. Dyeings cannot be discharged. It is also of interest for dyeing polyamide fibres. Fastness figures on wool include— light 5, washing (S.D.C. No. 2) 2–3, perspiration 4 duller.

Farbwerke Hoechst A.G.

JIG DYEING OF REMAZOL DYESTUFFS AT ROOM TEMPERATURE ON MERCERISED TEXTURED COTTON— This card contains dyeings in three depths (except black)

of 11 of the Remazol range of reactive dyes, together with 20 mixture dyeings. All these dyes, with the exception of Yellow G, Red 3B, and Brilliant Blue R, which give weaker dyeings than at 40–60°C, may be dyed at 20–30°C. The dyebath is set with 1 g/l. Calgon, half the required salt and half the dye, and the material given one end. The remainder of the dye and salt is then added, and the caustic soda and trisodium phosphate are added in two halves after the second and third ends, respectively. After all the alkali has been added dyeing is continued for 2 h at 20–30°C. The dyeing is then rinsed in cold water, acidified with acetic acid, rinsed in hot water, and soaped in neutral detergent for 5–10 min near the boil. Alkali must not be added to the detergent liquor or loss in depth of colour will occur.

J. R. Geigy A.G.

IRGALAN GREY BRL— This neutral-dyeing metal-complex dye gives redder greys than the Grey BL brand, and has better migrating power than the latter, especially in presence of ammonium sulphate. It is thus of particular interest for shading at the boil and for dyeing tightly woven wool cloths. It is also of interest for dyeing polyamide fibres. The fastness properties of the dyeings are of the usual high standard associated with this range and on wool include— light 6–7, washing *b* (8SI) 5, alkaline milling *b* (8SI) 4–5.

Rohner Ltd.

SINAGEN DYESTUFFS— This card contains prints on cotton cloth of 14 stabilised azoic combinations. The method of stabilising the diazo components enables development to be carried out by neutral steaming, thus avoiding the complications that arise when acid steam must be used and for which specialised equipment is needed. The hue and fastness characteristics of the dyeings obtained are the same as those given by the Ronagens, which are stabilised combinations based on the same components. Development in acid steam is, of course, possible. The dyes can also be developed by treatment at the boil in a bath containing acetic acid, formic acid, and Glauber's salt. Some may even be developed by simple exposure to direct sunlight for ½ to 1 h. When used in combination with resin-bonded pigments, dry-heating for 5 min at 140°C is adequate for development, glycerol being added to the paste.

Sandoz A.G.

SOLAR, CUPROFIX AND CUPROFIX-C DYESTUFFS STABLE TO CREASE-RESISTANT FINISHES— This card describes the effect of a representative range of resin finishes on the hue and fastness characteristics of 46 direct dyes dyed on cotton and viscose rayon. The finishes on viscose rayon, details of which are given, include Finish EM (Sandoz), Tootal Process (Tootal Broadhurst Lee), Aerotex Cream 450 Process (American Cyanamid), and Resloom M–75 Process (Monsanto). On cotton, a combined finish based on Finish EM and Ratifix A has been used. In each case, treated patterns are mounted alongside an untreated pattern to indicate the difference, if any, in hue and figures for daylight fastness, and the duration of exposure in the Fade-Ometer test (AATCC) is appended alongside. Since the effect on hue and light fastness is largely determined by the resin content of the material and this may vary with the type of fabric, the changes shown and the ratings given are intended only as an approximate indication.

THE WOOL RESERVE OF FORON DYESTUFFS— This is an appendix to pattern card No. 1298, *Foron Dyestuffs*. It contains dyeings of 14 Foron disperse dyes on Dacron and the corresponding stainings on wool cloth, before and after a clearing treatment with Resocol V, Arostit ZET, and formic acid, plus, in four instances, a final treatment in hydrogen peroxide added to the last rinse water.

CUPROFIX BLACK C-FBL— This direct black is very soluble and thus very suitable for padding and the Pad–Roll process. When aftertreated with copper sulphate, Cuprofix SL, or Resofix CU, it gives dyeings of good wet fastness which are stable to crease-resist finishing. It is also suitable for high-temperature dyeing. Fastness figures for a dyeing aftertreated with copper sulphate–acetic acid include— light (daylight) 6, washing (40°C ISO) 4–5, perspiration (ECE) 5.

DRIMARENE TURQUOISE Y-G: DRIMARENE NAVY Y-BL BY THE EXHAUSTION DYEING METHOD— This card describes the application of these two reactive dyes to cotton and viscose rayon by the exhaustion method in a short liquor on the jigger and in longer liquors in the winch or circulating-liquor machine. Dyeing and fixation are carried out at 95–100°C, using soda ash or trisodium phosphate as the alkali.

LANASYN CARBON BL— This neutral-dyeing metal-complex dye gives blacks on wool, silk, and polyamide fibres. Fastness figures on wool include— light (daylight) 7–8, washing (60°C) 4–5, severe milling 4–5.

SOLAR DARK BROWN 3LB: CUPROFIX DARK BROWN 3LB: CUPROFIX PRINTING BROWN 3LB— This direct dye gives bluish browns of good fastness to light and also to wet treatment when aftertreated on the fibre with either Sandofix WE or Cuprofix SL. It is stable to crease-resist finishing and is particularly recommended for dyeing furnishings. A concentrated brand is of interest for direct printing. Fastness figures for a direct dyeing on cotton include— light (daylight) 6, washing (40°C ISO) 3–4.

LYOGEN WD— This is a levelling agent specifically recommended for addition to the dyebath when dyeing neutral-dyeing acid and chrome dyes. It enables dyes normally applied from a neutral bath to be applied at pH 4–5 in presence of acetic or phosphoric acid, under which conditions brighter dyeings are obtained, since there is no yellowing of the wool. Dyeings free from skitteriness are obtained. Lyogen WD exerts a considerable retarding action; this is reduced by anionic compounds such as Sandozol N, which may be added to reduce the effect of the presence of too much retarding agent. The retarding and levelling action of this product is also of value when dyeing polyamide fibres and silk with these dyes. It is not recommended for use with neutral-dyeing metal-complex dyes.

Yorkshire Dyeware & Chemical Co. Ltd.

SERISOL AND SERILENE DYES ON POLYESTER SLUBBING— This card contains dyeings on Terylene slubbing, comb top (4 den., 3½–4½-in. staple, quality WI) produced by dyeing for 30 min at 130°C, at a 20:1 liquor ratio, in presence of 1 g/l. Dyapol PT. The dyes used include 17 Serisol disperse dyes and 19 disperse dyes especially recommended for use on Terylene and marketed as Serilene dyes. Among the latter is Serilene Azoic Black B2, a mixture of a diazotisable base and coupling component for producing fast azoic blacks. It is claimed that this is quicker and more convenient to use than the separate components. Where dyes are mixtures this is indicated in the description of the more important features and uses of each dye listed under each set of patterns. Supplementary information of particular interest to yarn and cloth dyers includes details of jig dyeing in presence of Dicene 60 as carrier, and the high-temperature dyeing of yarn in package form and cloth on the beam.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Modern Production Machinery for Non-woven Fabrics

R. R. Clegg *Text. Merc.*, **144** (6 Jan 1961) 14–16, 18–20

A review of the machinery and methods now in use. C.O.C.

Bibliography on Drying

P. Quensel *SVF Fachorgan*, **15** (Oct 1960) 622–623

A list of references to papers in American, British, French, German, and Russian technical journals on drying methods (15) and drying machines (18). L.A.T.

PATENTS

Wet Treatment of Loose Fibres and Hanks

Fleissner & Sohn Maschinenfabrik *BP* 851,644 (Germany 29 Oct 1957)

The materials are held on to the surface of a rotating horizontal drum by strong suction produced by a pump inside the drum acting upon liquor in which the drum is wholly or partly immersed. Means are provided to ensure that the flow of liquor into the drum is solely through the material being treated. C.O.C.

Stop for Milling Machine

A. Bertoldi *BP* 857,000 (Italy 24 May 1958)

A feeler in contact with the fabric in the machine detects knotting or entanglement of the fabric. It operates a pressure fluid control means which applies fluid pressure to separate the milling rollers and so stop the machine before the knot or entanglement reaches them. C.O.C.

Removing Backward Bow in Fabrics

John Dalgleish & Son *BP* 854,075 (8 July 1958)

A simple device for correcting backward bow in the weft of woven fabrics or in the courses of knitted fabric as it moves between let-off and take-up mechanisms. C.O.C.

Apparatus Resistant to Acid Chlorite Solutions

Deutsche Gold- und Silber-Scheideanstalt *BP* 852,900 (Germany 10 Jan 1957)

Modification of *BP* 817,615 (J.S.D.C., **76** (1960) 190). Aluminium–magnesium alloys coated by the eloxal process are resistant to acid chlorite and chlorine dioxide solutions in absence of nitrate or phosphate ions. C.O.C.

Centrifugal-space Hank Dyeing Machine

Hickory Dyeing and Winding Co. *USP* 2,922,696 (4 Aug 1954)

A centrifugally operating machine which dyes different areas of hanks in different colours so that they join each other without any overlapping and without leaving any undyed area between them. C.O.C.

Machine for Printing on Textiles, Paper, Plastics, Rubber, etc.

M. Stein and L. F. Werner *USP* 2,928,340 (21 Mar 1957)

A nearly completely automatic machine comprises a series of driven hollow drums each separately mounted and each carrying a flat rectangular silk screen held in cylindrical form. Each drum rotates its screen in contact along a tangent line against the material being printed. Each drum automatically raises and lowers its screen at least once during each revolution. Within each drum there is a distributor head for the printing paste or ink. This head does not rotate with the drum. The flow of paste or ink from this head is automatically turned on or off depending on whether the screen is in the printing or non-printing position. When the drum is in printing position the distributor head presses against the inner surface of the screen and acts as the squeegee. C.O.C.

Automatic Stencil Printing Machine

C. Masson *BP* 855,697 (France 16 Dec 1957)

The machine comprises a printing table and hinged stencil frames so mounted that they can be pivoted on to or away from material placed on the table. The frames are raised and lowered in unison by mechanism placed above the table. Another mechanism, also placed above the table, operates in unison the reciprocable squeegees for the stencils. C.O.C.

Sock-printing Machine

Burlington Industries *USP* 2,927,526 (2 Feb 1956)

A machine which prints a multicoloured pattern on to both sides of the sock at once. It operates continuously and at high speed, and delivers dried and printed socks ready for final packing. C.O.C.

Calenders for Laminating Thermoplastic Webs

Dornbusch & Co. *BP* 852,077 (Germany 14 Nov 1956)

Several roller sets are arranged one behind the other. Each set has a heating roller and a device for feeding a separate web and pressing it against the hot roller. Each set except for the first includes a counter roller forming with the hot roller a nip through which the web or webs from the preceding set, together with the web being fed to this set, are passed. C.O.C.

Web-coating Apparatus

Kodak *BP* 855,518 (U.S.A. 14 Dec 1955)

In a machine in which the applicator roller is partly immersed in a coating solution contained in a hopper, the bottom of the hopper is pitched to form an elevated ridge extending at right angles to the axis of the applicator roller and sloping away in both directions from a point opposite the middle third of the length of the applicator roller. There is an outlet for the coating solution in the bottom of the hopper adjacent to the ridge. This prevents solid particles in the solution from being transferred to the applicator roller and also ensures continuous stirring of the solution in the hopper.

BP 855,539 (14 Dec 1955)

Fixed helical partitions placed longitudinally in the bottom of the hopper intercept that part of the solution which is moved by, and counter to, the rotation of the applicator roller and cause the solution to move from the inlet to the outlet end of the hoppers. C.O.C.

Coating Webs with Viscous Aqueous Solutions

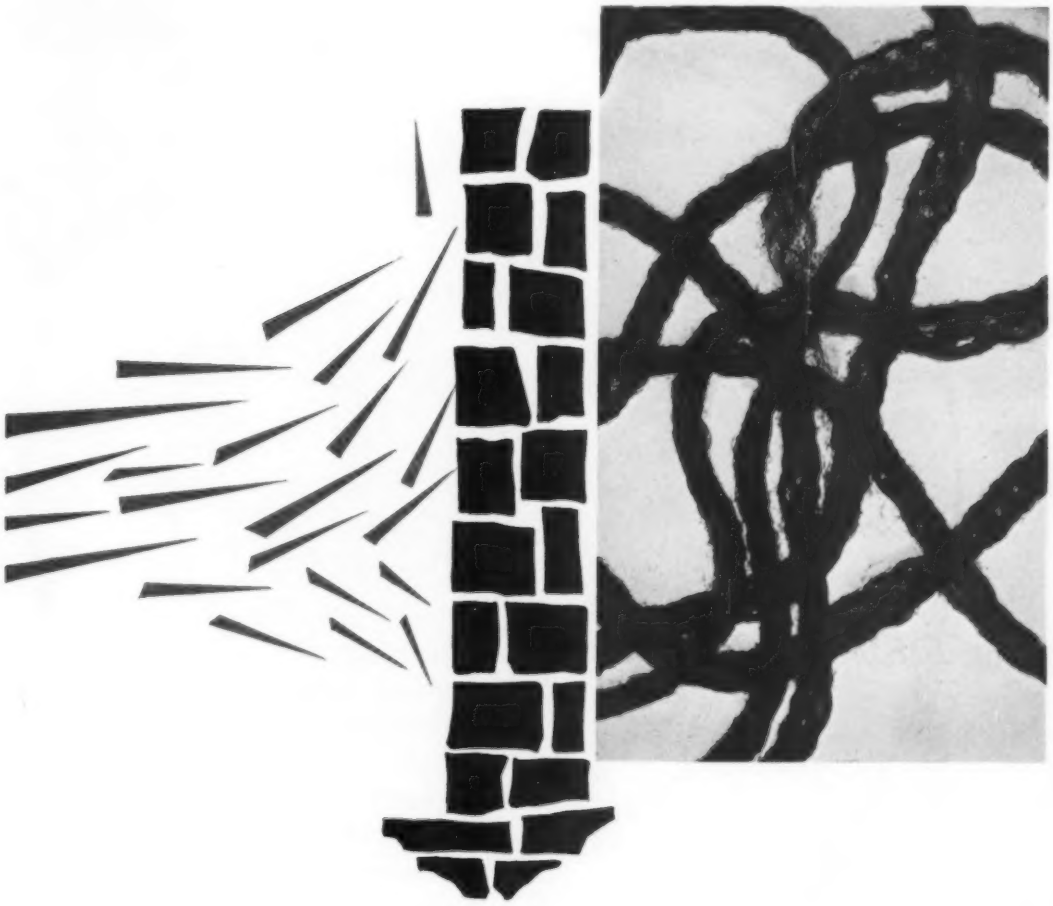
DuP *BP* 855,164 (U.S.A. 21 Aug 1957)

A layer of the solution is applied to a moving web. There is a device for reducing the thickness of this layer along a metering line transversely to the direction of travel of the web. An air knife doctors the layer along an impingement line transversely to the direction of travel of the web. The metering device is so made and placed that it prevents both the excess coating removed by the air stream and the air from passing it. This yields a coating of excellent smoothness and uniformity together with increase in coating speeds of up to 300%. C.O.C.

Coating and Drying Viscous Liquids on Flexible Webs

DuP *BP* 855,216 (U.S.A. 8 Nov 1957)

When reduced pressure coating (i.e. coating and drying in chambers under reduced pressure) is carried out at high speeds, the coating material upon extrusion does not boil even at pressures much below equilibrium pressure. Thus there is an apparent (or observed) equilibrium pressure which is lower than the static equilibrium pressure and which decreases with increasing coating speed. This is so much the case that when a liquid composition is applied to a moving web in a low-pressure atmosphere below the static equilibrium pressure of the composition, the composition sets without external cooling promptly after it contacts the web. The solvent and/or other vehicle in the composition separates from the solute and/or solids of the extrudate and appears as a clear supernatant liquid on the coating. Thus without further treatment there results a coating whose surface is extremely smooth, uniform and resistant to abrasion. By keeping the coated material under low pressure the solvent volatilises off in a few seconds. C.O.C.



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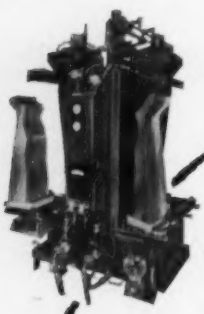

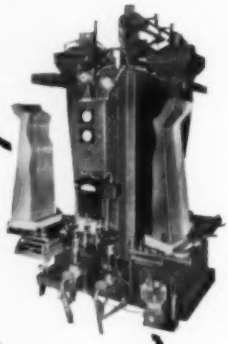

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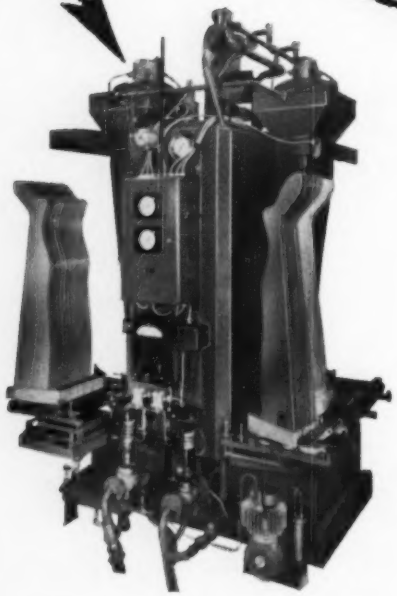

Temperature control can be remotely mounted (as illustrated) or mounted to individual machines

The features which make the GIRLAND PLASTICISER the most successful machine of its type—

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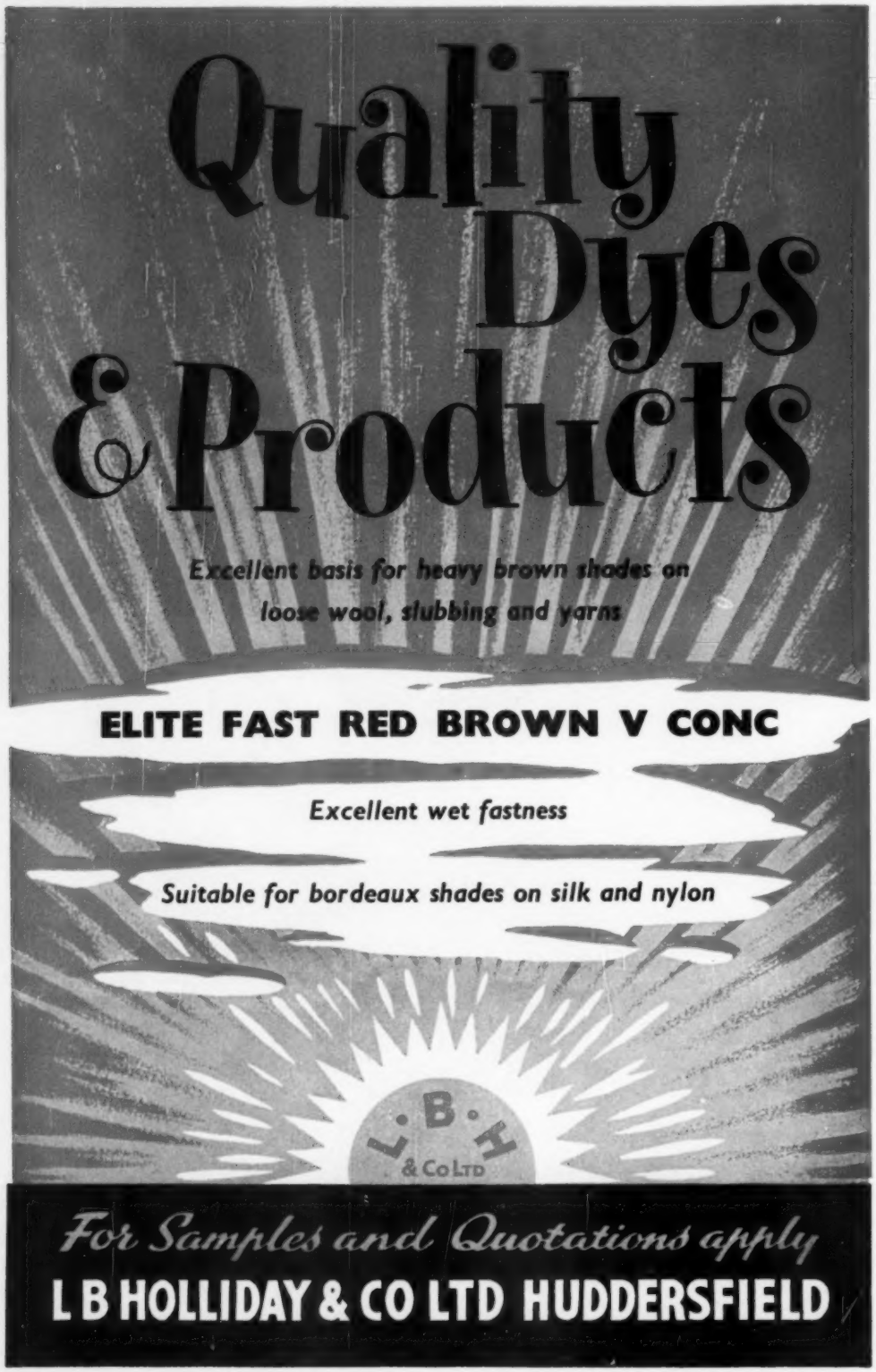
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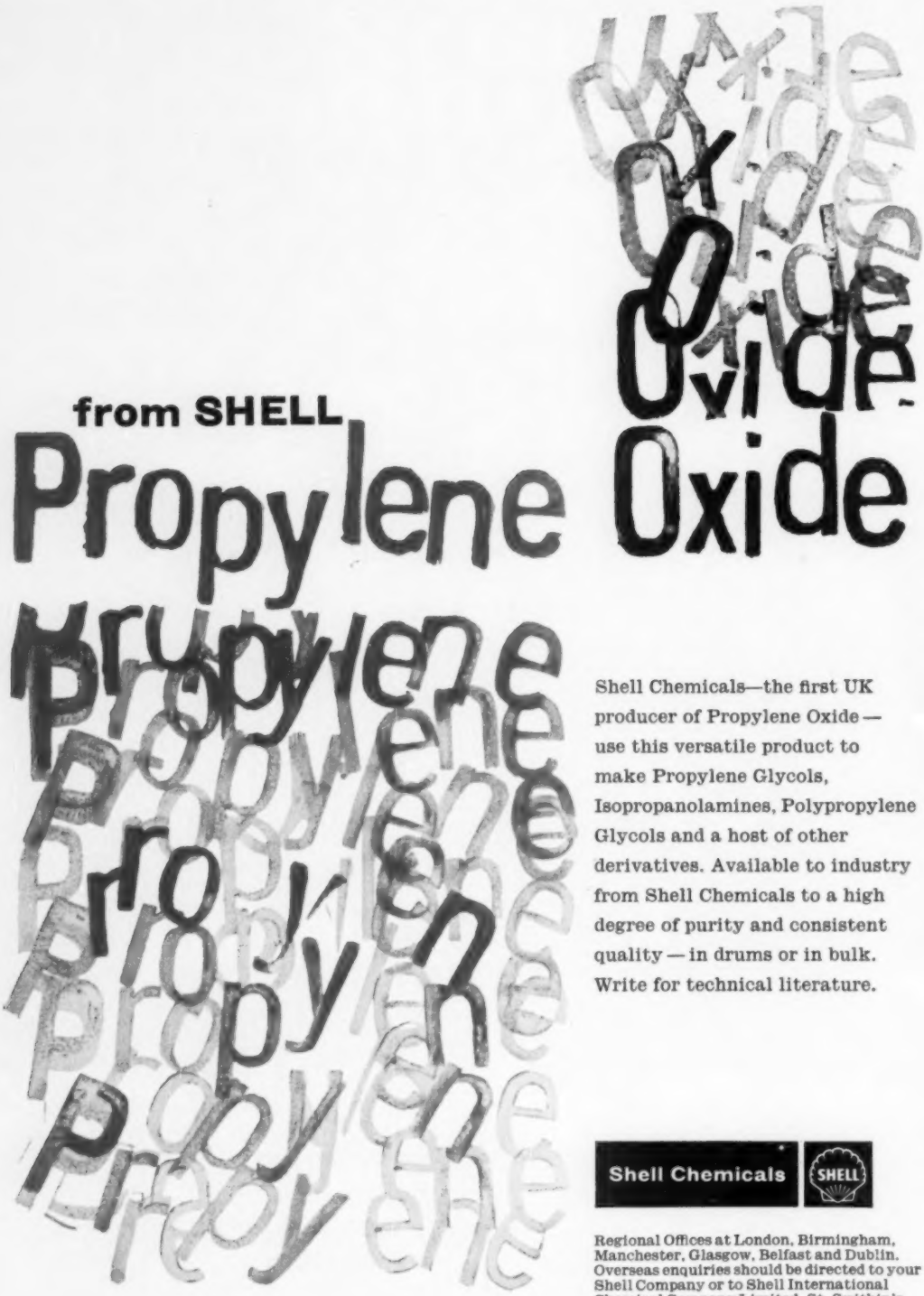

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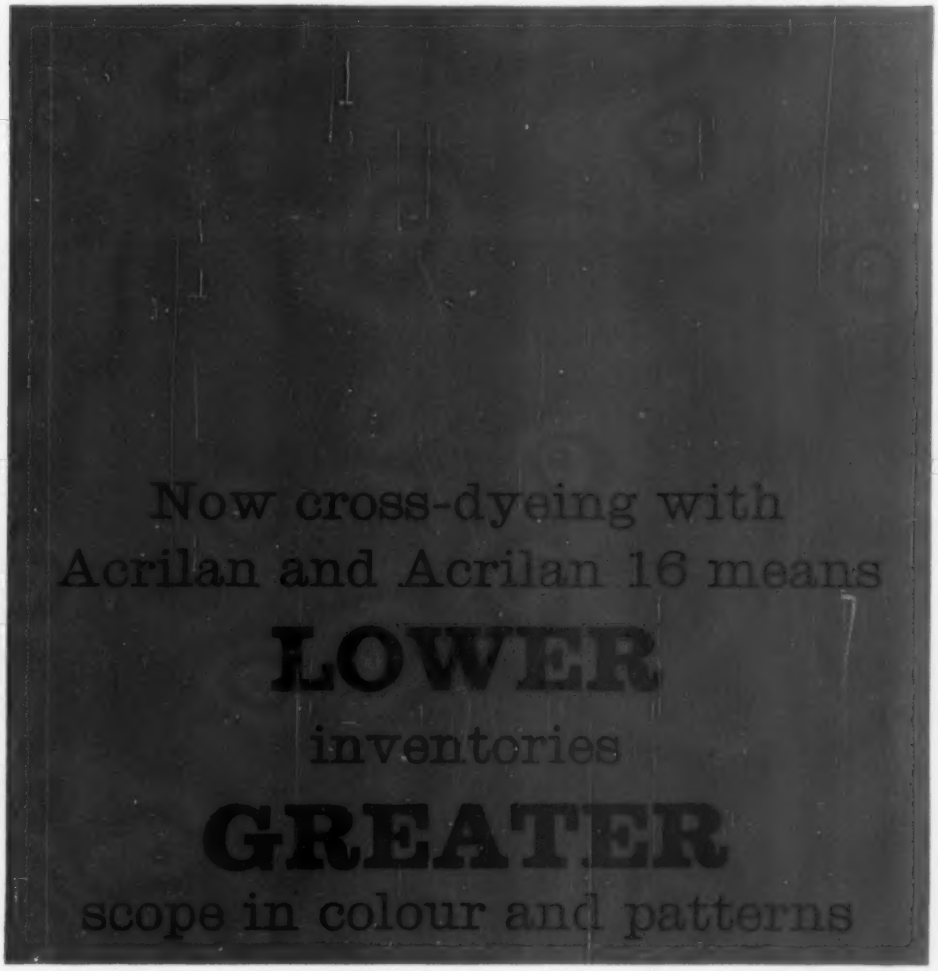

The introduction of Acrilan 16, with its different dyeing characteristics to regular Acrilan, offers new scope for cross-dyeing. Using blends of Acrilan and Acrilan 16, whether woven or knitted, you can piece dye a 100% Acrilan merchandise into a limitless range of two colour designs. To find out more about Acrilan and Acrilan 16, write or telephone:

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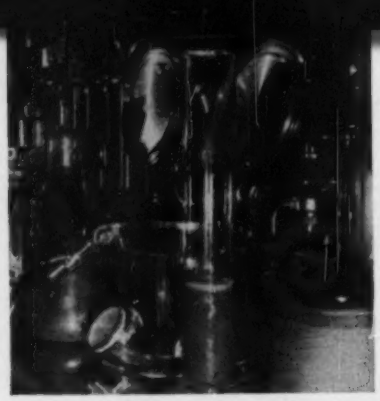

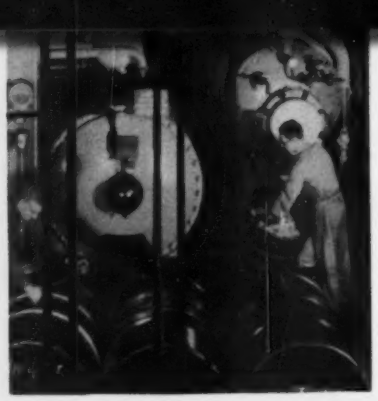

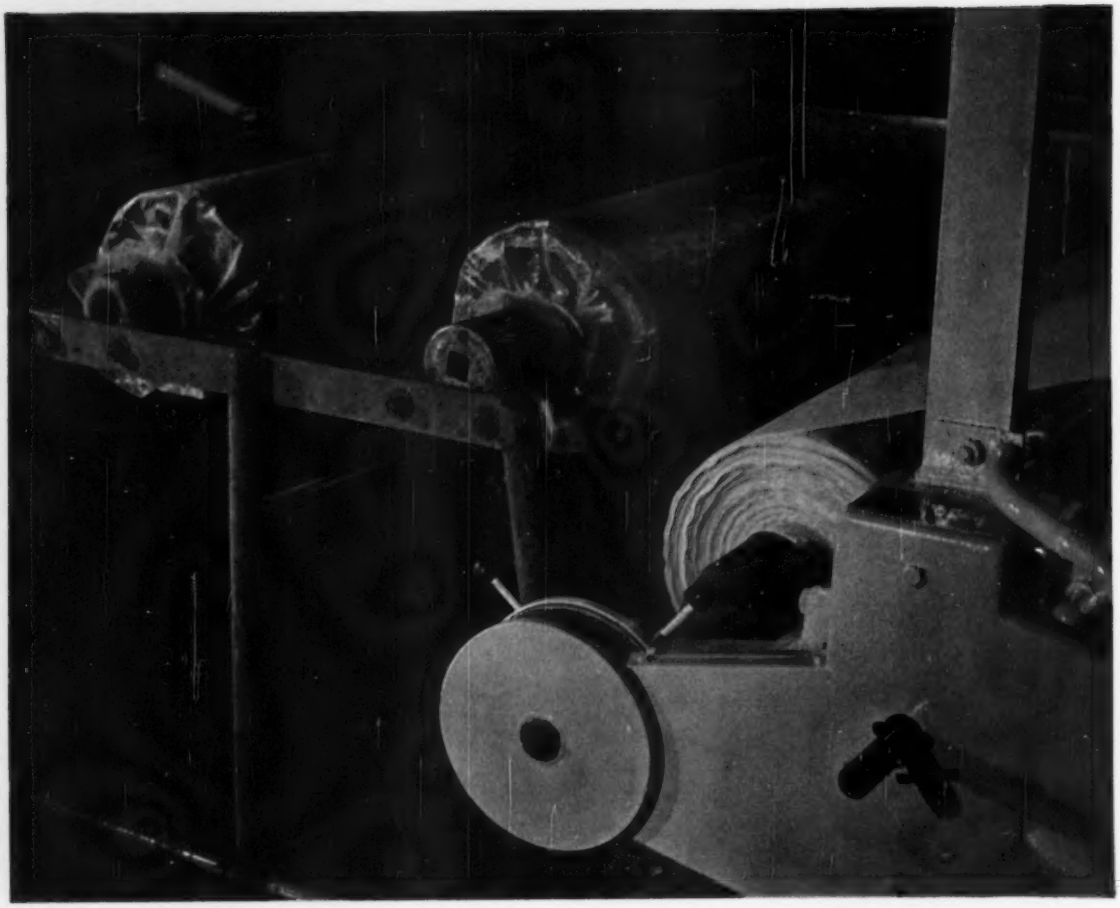

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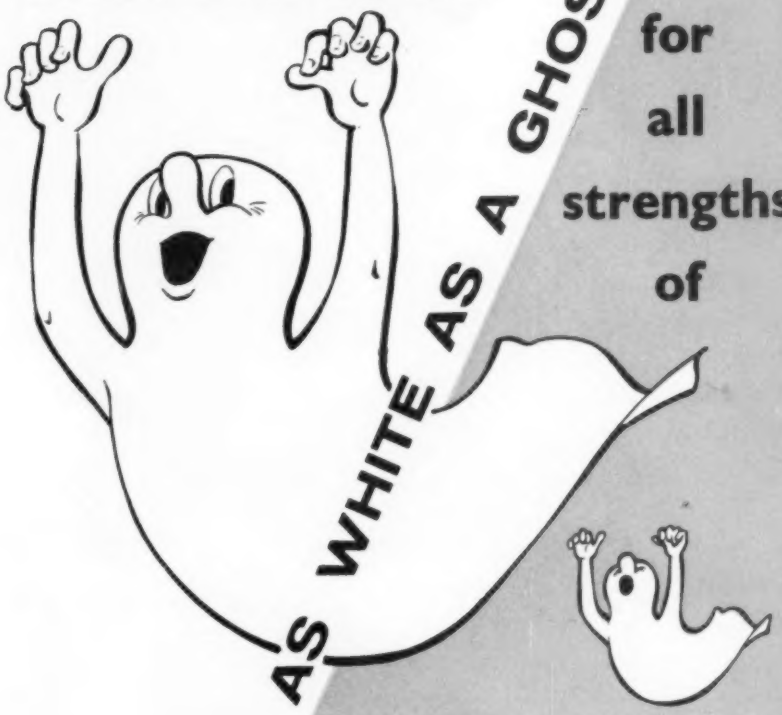

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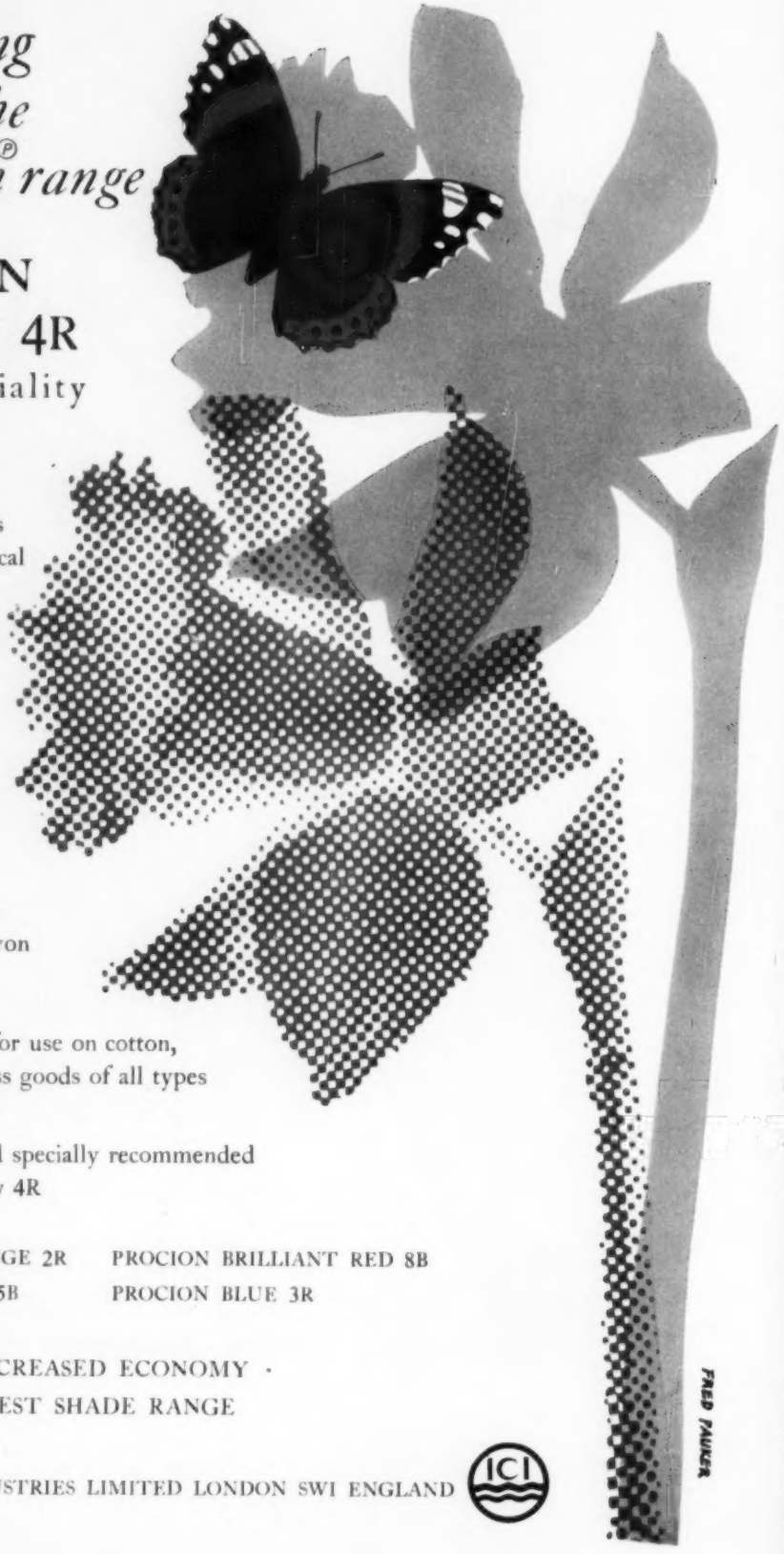

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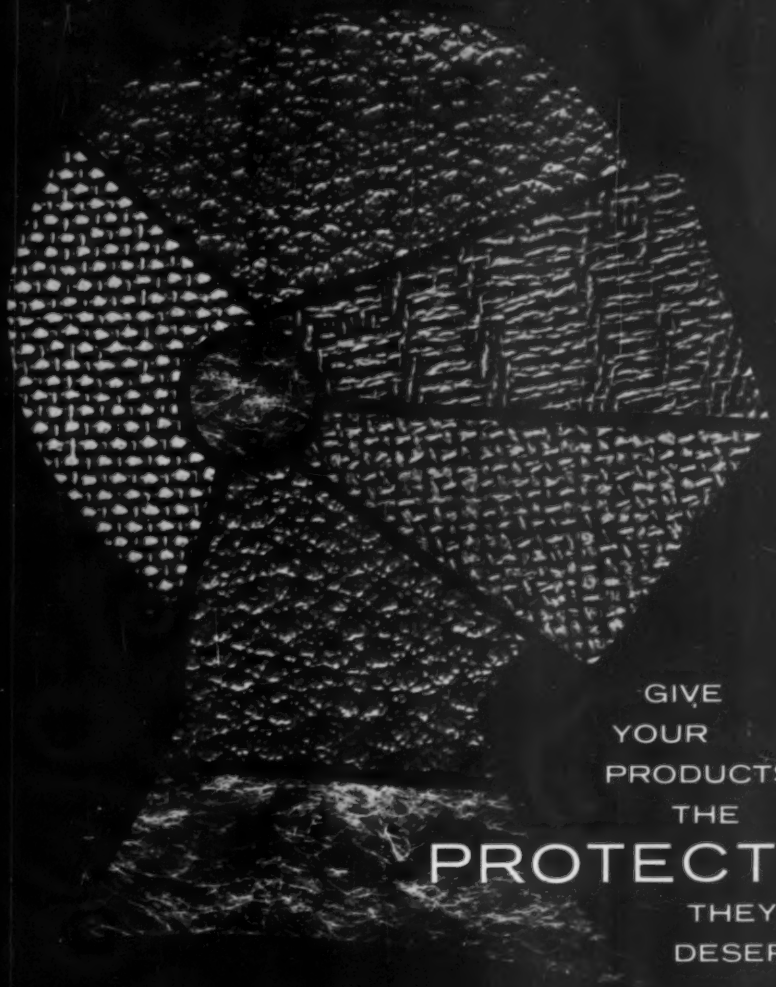

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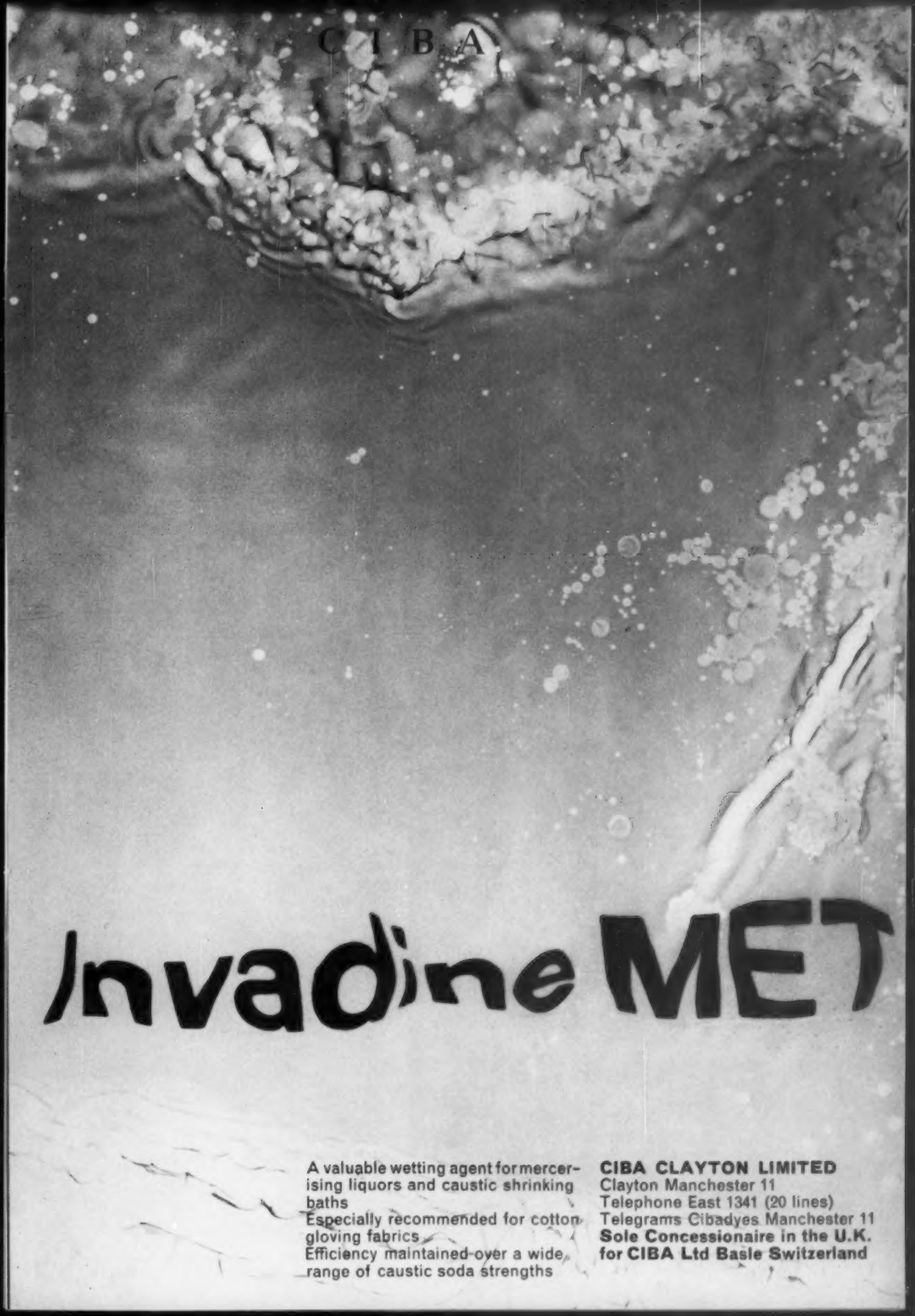

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simple reliable
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excellent solubility
easy wash-off of
unfixed dyestuff

for
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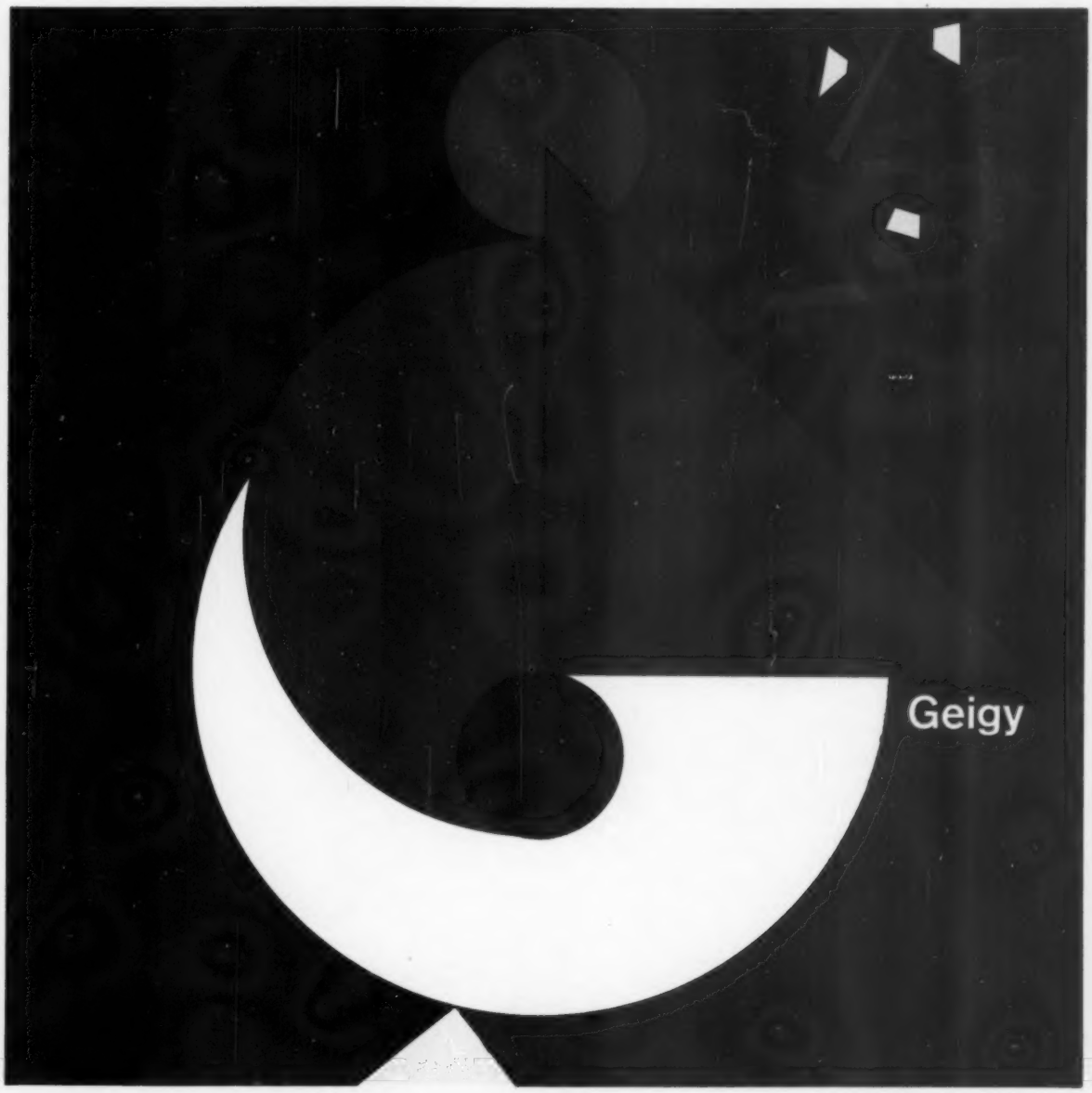

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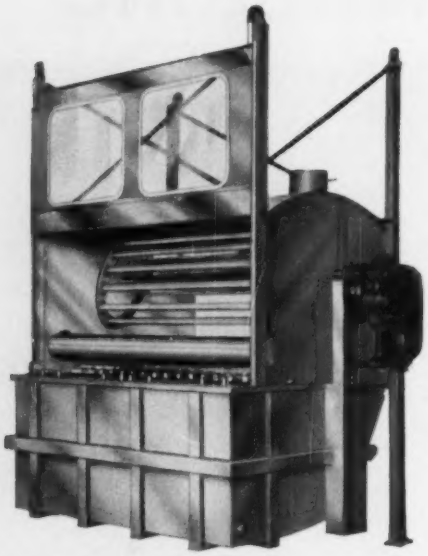

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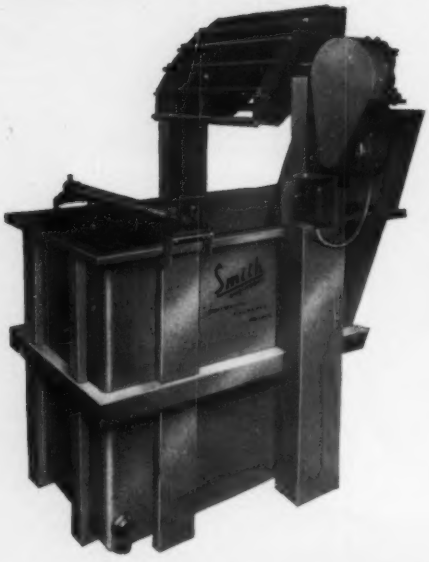

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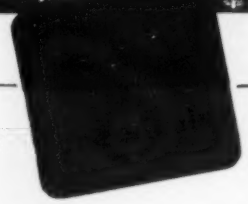

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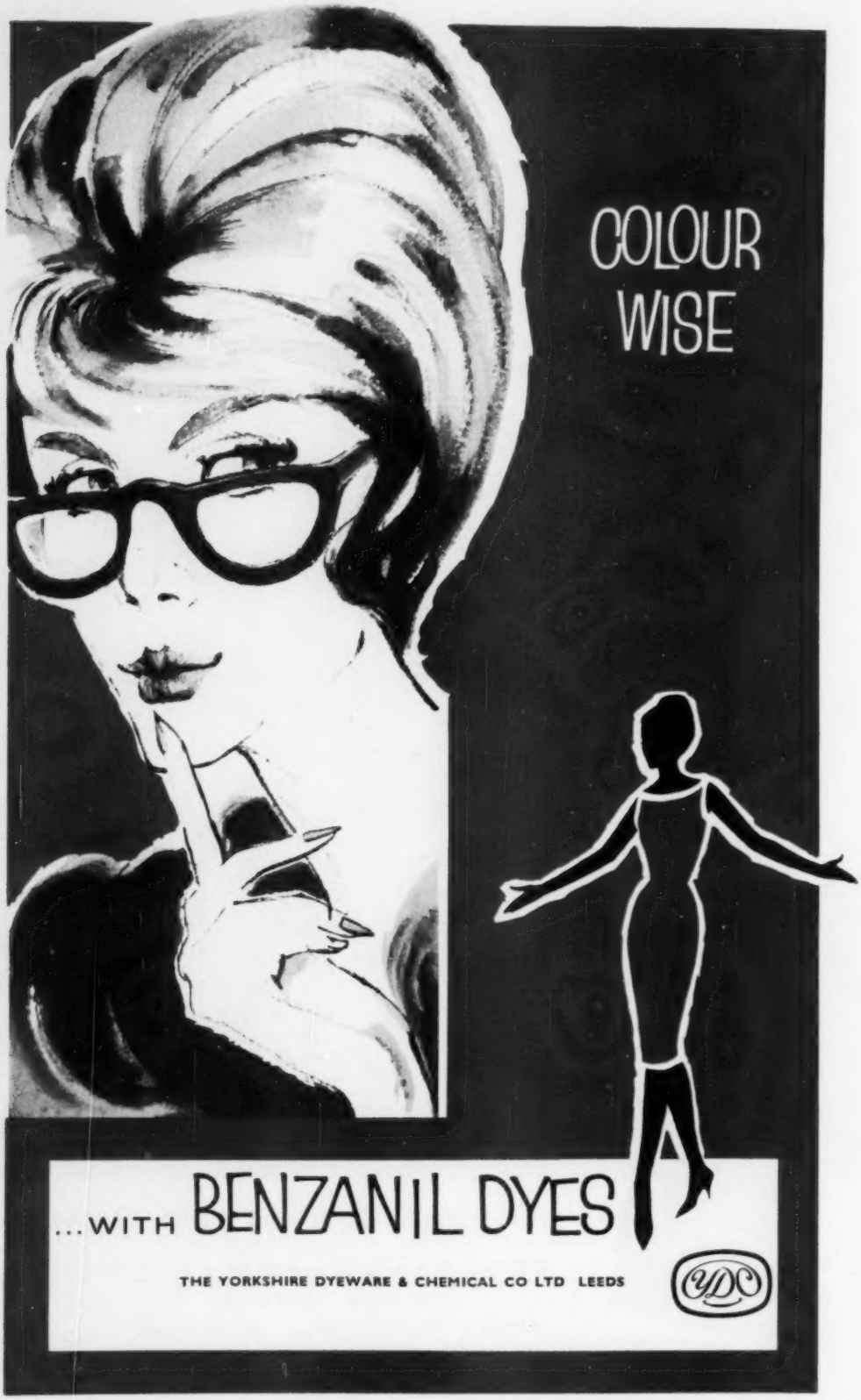
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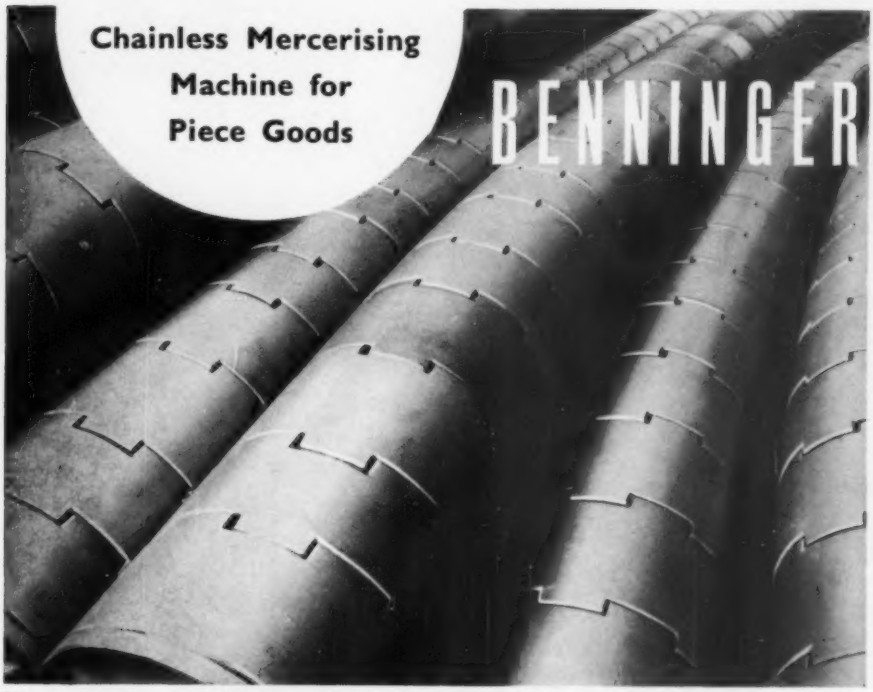

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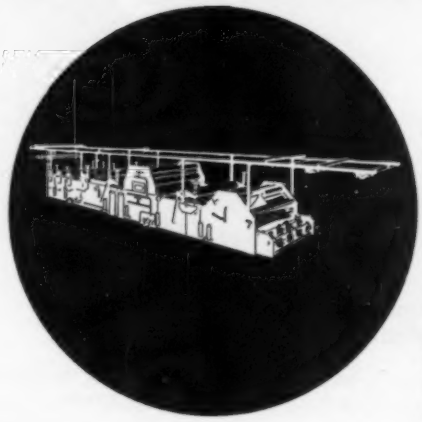

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These are some of the most important points expected to be accomplished by chainless mercerising machines. However, these can only be achieved if the cloth, in addition to being stretched longitudinally, is fully expanded widthways. In view of this fact we incorporate in our mercerising plant a special "Mycock" expanding device the efficiency of which has yet to be equalled. Indeed this device with its linked cast iron rollers and the clear separation of the cloth passages in an upper and a lower compartment has become a characteristic feature of our machine.

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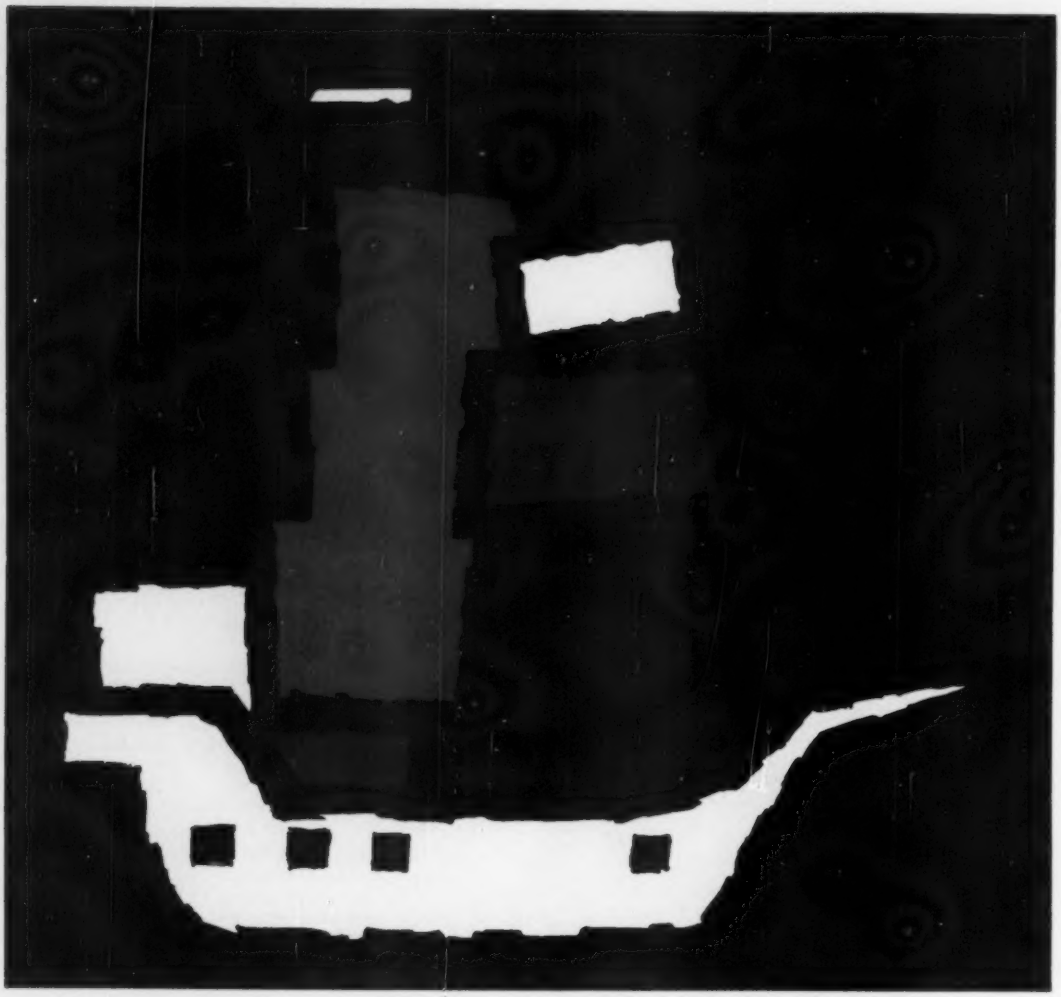

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Four-roll Calender

H. Burgtoff, E. Schwarz, J. H. Benecke, and O. Benecke *BP* 856,454 (14 June 1958)

A calender for rolling natural or synthetic material, laminating cloth, and rubber, etc. has its rolls placed in Z-formation. The two central rolls are mounted at each end in common slide blocks at each side of the calender. This permits the two central rolls to be raised and lowered in the common slide blocks and also moved so that, e.g., the left-roll ends are raised while the right-hand ends are lowered. This gives a slant relative to the roll mounted in front which can only be displaced horizontally and is not variable relative to the horizontal. C.O.C.

Nylon Hose Preboarding Machine

J. W. Lightburn & Son *BP* 844,972 (24 Nov 1958)

A preboarding machine for nylon stockings comprises a steam chamber into which two wheeled trays carrying hose forms are introduced alternately. Tray holders with wheel tracks are interlinked to swing alternately from a loading position alongside the chamber to a position in front of the chamber for introduction of the carriage. Means are provided for automatic locking and release of the trays and holders. G.E.K.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Some Industrial Uses of Enzymes

C. Boidin *Teintex*, **26** (15 Jan 1961) 33–45

Enzymes are first defined, classified, and their physical and chemical properties briefly described with relevant methods of analysis. An outline then follows of the respective uses of amylase, invertase, lipase, lipoxydase, pectinase, and protease, in the textile, bleaching, dyeing, cleaning, tanning, brewing, pharmaceutical, dairy, food, and fruit industries. H.H.H.

New Compound of Nicotinic Acid and its Bacteriostatic and Fungicidal Effects

J. Dick and J. Ristici

Studii şi Cercetări, Ştiinţe Chimice (*Timişoara*), **VII** (Jan–June 1960) 137–145

The preparation of mercury nicotinate (I) is described and data are reported with respect to its powerful bacteriostatic and fungicidal properties. Its use is based also on a new principle, viz. the promotion of growth with respect to the germination and development of the protected plant after the fungicidal activity has been exercised. This condition is fulfilled by hydrolysis of I to nicotinic acid, which is a growth promoter. Field experiments have confirmed this finding. H.H.H.

Compounds with Fungicidal Activity

G. Rossi and V. Corradini

Chim. e ind. (Milan), **42** (1960) 237

J. Amer. Leather Chem. Assocn., **56** (Jan 1961) 56

Compounds of formula $R^1R^1N{\cdot}CS{\cdot}SCH_2O{\cdot}COR^2$ and $R^1O{\cdot}CS{\cdot}SCH_2O{\cdot}COR^2$, where R^1 = Alk, R^2 = CH_3 or C_6H_5, obtained by reaction of an alkaline dialkyldithiocarbamate or xanthogenate with chloromethyl esters, e.g. acetate, benzoate, show noticeable fungicidal activity, especially against *Aspergillus niger*, *Penicillium Roqueforti*, and *Saccharomyces ellypsoideus*. The most active compound, $(CH_3)_2N{\cdot}CS{\cdot}SCH_2O{\cdot}CO{\cdot}C_6H_5$, has activity similar to that of Na pentachlorophenate. M.T.

Studies on Textile Finishing Agents. XVIII—Effects of Additives in the Polymerisation and Condensation of *N*-Methylolacrylamide within Cotton Fabrics

H. Kamogawa, R. Murase, and T. Sekiya

J. Soc. Textile Cellulose Ind. Japan, **16** (Oct 1960) 873–878

In the polymerisation and condensation of *N*-methylolacrylamide (I) in cotton, acrylamide (II) and hexamethylene diamine (III) hydrochloride were added to the treating baths to adjust the network structure of the polymer in the fibre. The optimum monomer mixing ratio was approximately 0·6 mol. I in II and 15% in III. Softening agents of low molecular weight, e.g. octadecyleneurea, were found to be most effective, as were polymer emulsions of high flexibility. Aftertreatment of I-treated (uncured) fabrics with III or HCHO was extremely effective in improving strength and crease-recovery. A.T.P.

Neutral Salt-free Basic Chromium Salts and Use of Anion Exchangers for their Preparation

K. H. Gustavson

J. Soc. Leather Trades Chem., **45** (Jan 1961) 9–16

Solutions of basic Cr salts, free from neutral salts, are of interest for, e.g. investigating certain aspects of the reaction of chromium complexes with proteins (e.g. chrome tanning). They may be prepared by gradual addition of an OH-charged, anion-exchanging resin of the quaternary-base type to Cr chloride, perchlorate, or sulphate soln. Extremely basic chlorides and perchlorates of Cr, with acidities of 25–35%, may also be prepared under favourable conditions, without appreciable loss of Cr; strongly basic anion exchangers are better for this purpose than weak ones. J.W.D.

PATENTS

Reducing Agents for Use in Vat and Sulphur Dyeing

BASF *BP* 855,494 (Germany 4 June 1958)

Level dyeing is obtained in a hank- or spray-dyeing machine by use as reducing agent of an alkali-metal or NH_4 salt of a linear or branched hydroxyalkylsulphinic acid of 2–4 C in which the OH and sulphinic acid groups are attached to the same C atom and/or an ammonia derivative containing at least once, directly attached to the N atom, the radical of an alkali-metal or NH_4 salt of a linear or branched alkylsulphinic acid of 2–4 C in which the N and S atoms are attached to the same C atom, e.g. Na hydroxyethane sulphinate or the product of the reaction between ammonia and the ammonium salt of 1-hydroxyethanesulphinic acid (I).

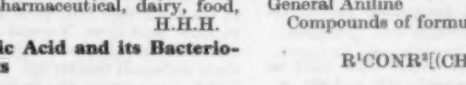
BP 855,496 (Germany 26 June 1958)

The above reducing agents give excellent results in continuous dyeing by the pad–steam process. C.O.C.

Amides of Aminoalkyl Pyrrolidones

General Aniline *BP* 860,655 (3 June 1959)

Compounds of formula—



(R^1 = aliphatic of 5–22 C; R^2 = H, Alk or hydroxalkyl of < 6 C; m = 0, 1, 2 or 3; n = 1, 2 or 3; $(CH_2)_n$ may be substituted by OH) are obtained by selected acylation of aminoalkylpyrrolidones with aliphatic or aromatic monocarboxylic acids. They have a wide field of application, e.g. as antistatic, antioxidant, and softening agents; in the preparation of thixotropic paints; as wetting, flushing, and grinding acids for pigments; in textile oil emulsions; in fat liquors; dyeing; mothproofing compositions, etc. C.O.C.

β-Aminoalkyl Carbamates—Plasticisers and Softening Agents, Components for Resins for Finishing Paper and Textiles

Dow Chemical Co. *BP* 861,045 (27 Feb 1958)

Compounds of formula $NH_2{\cdot}CHR^1{\cdot}CHR^2{\cdot}O{\cdot}CO{\cdot}NH_2$ (R^1 and R^2 = H or Alk of 1–4 C and together are of < 5 C and when R^1 = H then R^2 = Alk and *vice versa*), e.g. β-aminomethylethyl carbamate, are clear, colourless, viscous, and water-soluble. They are good plasticisers for resins, and softening agents for paper, regenerated cellulose film, etc. They form resins with formaldehyde which can be used to impart wet-strength to paper and as crease-shedding finishes for textiles. C.O.C.

Titanate–Silicone–Wax Compositions for Imparting Water Repellency

General Electric Co. *USP* 2,928,799 (13 May 1957)

A mixture of (1) 10–60% of a compound of formula $Ti(OR^1)_4$ (R^1 = aliphatic hydrocarbon of < 12 C or hydroxylated and aminated aliphatic hydrocarbon of < 12 C and < 4 OH) or its aliphatic hydrocarbon-soluble partial hydrolysate, (2) 25–75% of a methyl polysiloxane copolymer containing trimethylsiloxy and SiO_2 units and 1–1·25 CH_3 per Si, (3) 10–50% of a fluid organopolysiloxane of formula $(R^2)_aH_bSiO_{(4-a-b)/2}$ (R^2 = unsubst. monovalent hydrocarbon, a = 1·0–2·25; b = 0–1·25; $a + b$ = 1·96–2·25) of viscosity 5 to 1,000,000 cS at 25°C, and (4) a wax, renders porous materials water-repellent without requiring heat to develop the optimum water-repellency. C.O.C.

Bleaching Composition

Unilever *BP* 855,735 (9 May 1958)

Those acyl organoamides which, when heated in water with a persalt, yield a solution which liberates iodine from

KI at 0°C, are excellent activating agents for inorganic persalts used as bleaching agents at 50–60°C. An example is *NN*-diacetyl-*p*-chloroaniline. C.O.C.

Bleaching Compositions
Newlands Bros. & Mumford *BP* 860,651 (1 Sept 1958)
A mixture of Na_2CO_3 (48–54%), $NaClO_3$ (26–22·5%), $NaClO_4$ (21–17·5%) and Na persulphate (5–6%) is stable and can be stored and transported in perfect safety. When dissolved in water it renders chlorine and oxygen available for bleaching. C.O.C.

Mothproofing Agents
Gy *BP* 859,345 (Switzerland 9 Sept 1958)
Compounds of formula $Ar^1{\cdot}SO_2{\cdot}NR{\cdot}Ar^2$ (Ar^1 and Ar^2 = phenyl subst. by at least one trifluoromethyl and at least one Hal in either radical, both of which may be substituted by further Hal, NO_2 and/or Alk or alkoxy; R = H or Alk), e.g. 3-trifluoromethyl-4-chlorobenzene sulphonic acid-2′,4′-dichloro-5′-trifluoromethylanilide, give keratinous materials excellent protection against clothes moths and carpet beetles. They are applied from aqueous solution and give a mothproof finish of good fastness to washing. C.O.C.

Emulsifying Agents for Resins
Shawinigan Chemicals *BP* 856,920 (Canada 22 Sept 1958)
Water-soluble vinyl acetate–polyoxyalkylene glycol copolymers and their derivatives are good emulsifying agents for resins. Their use yields aqueous emulsions which form water-insensitive films. C.O.C.

Fungicides and Vulcanising Agents
Shell Research *BP* 857,166 (12 Mar 1958)
The product obtained by treating a solution of a compound of formula MS·SC·NH·R·NH·CS·SM (R = hydrocarbon or $-CH_2CH_2N(CS{\cdot}SM)CH_2CH_2$; M = salt-forming atom or group) at 30–60°C with cyanuric chloride or bromide has excellent fungicidal properties and can be used as a vulcanising agent. C.O.C.

Polar Brilliant Blue GAW (see C.I. Acid Blue 127) as Bluing Agent in Laundry Detergents (IV p. 179)
Thickening Agents for Printing Pastes Containing Reactive Dyes (IX p. 185)
A Study of Storing and Preservation Conditions of Wet Spruce Mechanical Pulp. I—Heat Inactivation of Phenylmercuric Acetate as a Preservative for Groundwood Pulp (XI p. 188)
Curing of Polyepoxides (XIII p. 189)

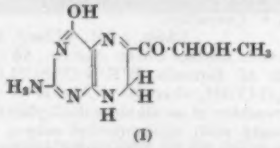
IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Autoxidation of Selected Organic Solids in Presence of Alumina
E. F. Pratt and L. E. Trapasso
J. Amer. Chem. Soc., **82** (20 Dec 1960) 6405–6408
Some organic solids have been autoxidised by passing air through a mixture of the powdered cpd. (0·01 mol.) and ordinary or sodium-methoxide-treated alumina (50 g) at temperatures above 55°C. Methylene and hydroxymethylene groups adjacent to aromatic rings have been converted to carbonyl groups in the presence of both kinds of alumina. Several additional types of autoxidation have been carried out under the same conditions. Cpd. examined include the following, the products being given in parentheses together with % yield from untreated and sodium-methoxide-treated alumina respectively—9-hydroxy-9,10-dihydroanthracene (anthraquinone, 60%, 78%), 9,10-dihydroanthracene (anthraquinone, 59%, 83%), anthracene (anthraquinone, 6%, 18%), 9-methylanthracene (anthraquinone, 20%, 0%), acridane (acridine, 50%, 78%), 2-naphthylamine (dibenzo[*a*,*h*]phenazine, 40%, 29%), and 9-chlorofluorene (fluorenone, 42%, 0%). The ability of the base to promote these reactions implies that such compounds react readily via ionic processes in the solid phase. F.J.

Dipole Moments and Sorption Characteristics at the Solution-Solid Interface of a Series of Substituted Azobenzenes
D. A. Ibbitson, T. Jackson, (Miss) A. McCarthy, and C. W. Stone *J.C.S.*, (Dec 1960) 5127–5132
Sorption of *p*-hydroxy-, 4-hydroxy-3-methyl-, and 2-methyl-, 2-hydroxy-5-methyl-, *p*-acetoxy-, *p*-amino-, and *p*-acetamido-azobenzene on activated alumina, provide evidence that hydrogen-bond formation is responsible for the sorption process. Standard thermodynamic free-energy changes, calculated from the isotherm data, show that the hydroxyazobenzenes (I) have a greater affinity for the alumina surface than *p*-aminoazobenzene (II). Sorption saturation values are explained in terms of size and orientation of the sorbate mol., and it is suggested that I may be sorbed on different surface sites from II. That the dipole moments of 2-hydroxy-5-methylazobenzene in benzene and dioxan are almost the same is considered to be evidence of intramol. H-bonding in this cpd. Dipole moments of the azo cpd. in benzene are compared with those of the corresponding benzene deriv., and explained by the difference between the $+M$ effects of the amino- and the hydroxy-group. The monosubstituted cpd. used above are of interest because of their previous use for the determination of an arbitrary gradation of activities of chromatographic alumina.
H.H.H.

Lightfastness and Surface Activity of Dyes: Effect of Alkylation and Sulphonation
C. H. Giles, G. Baxter, W. A. Black, N. Macaulay, and S. M. K. Rahman
Text. Research J., **30** (Dec 1960) 934–943
Several series of dyes of the sulphonated anthraquinone, sulphonated and unsulphonated azo, azomethin, and merocyanine classes were irradiated in transparent films and their fading rates and characteristic fading curves determined. Marked increase in surface activity of an ionic dye tends to decrease light fastness in normal substrates. A smaller increase in surface activity, e.g. by introducing a short alkyl chain, usually increases light fastness. The orientation of a number of sulphonate groups affects light fastness. More even distribution of such groups or increase in their number improves light fastness: this rule applies to most acid dyes. Normal aggregation of the dyes in the substrate is sufficient in most cases to mask differences in their chemical reactivity. The less soluble a dye is in the film before setting, the higher the fastness to light, and the more soluble it is the lower the fastness. Molecularly dispersed dyes, e.g. those in the merocyanine series used in collodion, have the same fastness even when there are marked differences in length of an attached alkyl chain.
C.O.C.

Structure of the Yellow Pigment from *Drosophila*
S. Nawa
Bull. Chem. Soc. Japan, **33** (Nov 1960) 1555–1560
The structure (I), 2-amino-4-hydroxy-7,8-dihydro-6-lactylpteridine, is proposed for the yellow pigment Sepiapterin occurring in the eyes of *Drosophila melanogaster*.



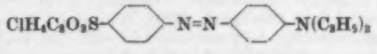
(I)

The evidence for this is discussed. It is based on the results of periodate oxidation, photodecomposition, and the fact that air oxidation in borax solution was found to yield one mole each of lactic acid and 7,8-dihydroxanthopterin, accompanied by the uptake of 0·5 mole of oxygen.
I.G.L.

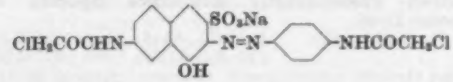
Reactive Dyes
V. F. Borodkin *Tekhnol. tekstil prom.*, (3) (1960) 102–105
Chem. Abs., **54** (25 Nov 1960) 25839
Study of the possible reactions between various fibres and simple azo dyes containing chloroacetamide, chloroethylsulphamoyl, and 2-chloro-3-hydroxypropyl groups. Dyes containing the chloroethylsulphamyl group gave dyeings on cotton and silk of good fastness to washing, perspiration, and dry rubbing, and satisfactory fastness to wet rubbing. Some of the other dyes had good to excellent fastness to light on cotton, nylon, and wool. C.O.C.

Reactive Dyes
T. Kitao, N. Kuraki, and K. Konishi
Bull. Univ. Osaka Prefect., Ser. A, **7** (1959) 85–101
Chem. Abs., **54** (25 Nov 1960) 25836
19 Water-soluble direct dyes (I) containing the vinyl

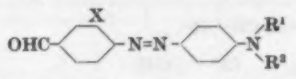
sulphonyl group (II), stabilised as the 2-chloroethylsulphonyl group (e.g. sodium 7-amino-4-hydroxy-8-[(*p* - chloroethylsulphonyl)phenylazo] - 2 - naphthalenesulphonate, dyeing yellowish red on silk, nylon, cotton, and Vinylon) were prepared and were fast to light (except some on nylon), rubbing, and washing. 10 Disperse azo dyes containing II were all fast-dyeing, especially on nylon. Thus, the yellowish orange—



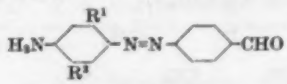
Diazotisation of *p*- and *m*-$H_2N{\cdot}C_6H_4{\cdot}SO_2C_2H_4Cl$ and coupling to various Naphtols gave 11 dyes, fast to light, rubbing and washing. 18 Water-soluble dyes similar to I but containing chloroacetyl groups were synthesised, e.g.



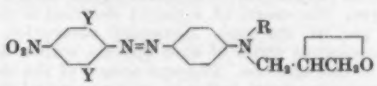
(dark red on silk, reddish brown on nylon). They reacted well with silk and nylon, but not as well with cotton, and had good fastness. When $-COCH_2Cl$ was replaced by $-COCH_2{\cdot}CH_2Cl$ (III), the dyes reacted well with silk and nylon, but poorly with cotton. Dyes containing $-COCHCl{\cdot}CH_3$ were inferior to those containing III, but dyes with $-CO{\cdot}CHCl{\cdot}CH_2Cl$ were better. 6 Disperse dyes—



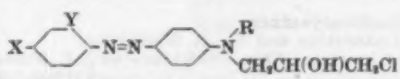
X = H or NO_2 (e.g. $R^1 = C_2H_5$, $R^2 = C_2H_4OH$, X = NO_2, orange) and 3 dyes—



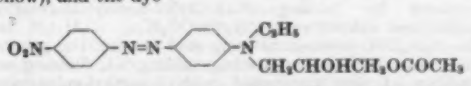
(e.g. R^1 = OCH_3, R^2 = CH_3, yellowish orange) had excellent washing and rubbing fastness on Vinylon but poor light fastness. The light fastness of the last three was improved by diazotisation and coupling on the fibre to give violet to olive dyes. 4 Disperse dyes—



(Y = H, R = CH_3, yellowish red; Y = H, R = C_2H_5; Y = Cl, R = CH_3; and Y = Cl, R = C_2H_5) and 5 of type—



(e.g. X = $NHCOCH_3$, Y = H, R = C_2H_5, reddish yellow), and the dye—



all dyed fast shades on the four above fibres. After Vinylon was subjected to acetal exchange with *m*-aminobenzaldehyde, extraction with acetone, diazotisation and coupling to azoic coupling components gave, e.g. a dark red with C.I. Azoic Coupling Component 7 (similarly dyes from 2-chloro-*p*-aminobenzaldehyde). A.T.P.

Separation Components in the Synthesis of Azo Dyes XIV—Determination of Absorption Spectra in the Triazine Series

J. Reichel and A. Demian

Studii şi Cercetări, Ştiinţe Chimice (*Timişoara*), **VII** (Jan–June 1960) 99–112

The preparation of three unsym. green azo dyes is described. Each has a triazine cpd. as middle component combined with the blue and yellow end components employed previously (cf. *J.S.D.C.*, **76** (Nov 1960) 636). After purification by paper chromatography, the separation effect of the central triazine system was established by measurement of absorption spectra. Comparison is made with the separation effects of other central components. H.H.H.

The Colour Behaviour of 6-(2-Hydroxy-3-naphthamido)-1-naphthol-3-sulphonic Acid

J. Reichel and D. Purdela

Studii şi Cercetări, Ştiinţe Chimice (*Timişoara*), **VII** (Jan–June 1960) 129–136

Coupling of the now readily accessible title intermediate with diazotised amines affords excellent yields of monoazo dyes with the azo link to the aminonaphthol residue, and also disazo dyes. On account of their sparing sol., the direct development of these dyes on the fibre is recommended. Fixation of the sulphonated azo-naphthols, and of the monoazo cpd., follows on cotton by treatment with alkali carbonate soln. and on wool with acetic acid. Subsequent coupling on the fibre with different diazotised amines affords developed dyes of particularly beautiful hue and relatively good fastness. H.H.H.

Azo Dyes based on Dimethyl Esters of Aminophenylsulphonamidophosphoric Acids

A. V. Kirsanov and N. G. Feshchenko

Zhur. obshch. khim., **30** (Oct 1960) 3389–3392

Azo dyes of formula $ArN{=}NC_6H_4SO_2NHPO(OCH_3)_2$ (I) are obtained by coupling the diazonium salts of the 3 isomeric dimethyl esters of aminophenylsulphonamidophosphoric acid ($NH_2C_6H_4SO_2NHPO(OCH_3)_2$) with phenol, β-naphthol, and dimethylaniline. These dyes are acid–base indicators. I (Ar = *p*-$(CH_3)_2NC_6H_4$–) has the same pH range as Methyl Orange but the colour change at the transition point (pH 4) is sharper. The latter dye is synthesised alternatively from the parent sulphonamide by the successive action of PCl_5 and methanol ($-SO_2NH_2 \rightarrow SO_2N{=}PCl_3 \rightarrow -SO_2NHPO(OCH_3)_2$). The phosphazine reaction is thus made applicable to compounds containing azo and dimethylamino groups. Physical and chemical constants of the above 9 dyes are tabulated. G.J.K.

Condensation of Naphthalic Anhydride and its Derivatives with Arylamines. III—Azo Dyes, Derivatives of the Phenylimide of Naphthalic Acid

B. M. Krasovitskiĭ and E. E. Khotinskaya

Referat. Zhur., Khim., **1957**, Abstr. No. 30587

Chem. Abs., **54** (25 Nov 1960) 24579

Azo dyes derived from diazotised 4-aminonaphthalic acid phenylimide (I) are deeper in colour than those from diazotised naphthalic acid *p*-aminophenylimide. Introduction of substituents such as Cl into the benzene ring of the imide, or of Br into the naphthalene ring, has little influence on the hue. Thus I diazotised and alkali-coupled with H acid gives dark violet. E.S.

Azo Dyes Derived from 2-Methylbenzimidazoles and 5-Nitro-2-methylbenzimidazoles

M. T. Le Bris, H. Wahl, and T. Jambu

Bull soc. chim. France, (1959) 343–348

Chem. Abs., **54** (25 Nov 1960) 24674

Diazonium salts couple with 1,2,3-trimethylbenzimidazolonium salts to give disazo compounds, both azo groups being attached to the C atom of the 2-methyl group. 1,3 - Dimethyl - 2 - (*p* - dimethylaminobenzalmethyl)benzimidazolonium iodide condenses with phenylhydrazine to give the monoazo compound 1,3-dimethyl-2-phenylazomethylene-2,3-dihydrobenzimidazole. The 5-nitro compounds behave similarly. E.S.

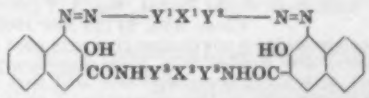
Synthesis of Azo Paints for Car Lacquers

T. I. Orlova and G. M. Makarovskaya

Khim. Prom., (1959) 582–584

Chem. Abs., **54** (25 Nov 1960) 25877

Describes in general terms, no specific data being given, pigments of formula—



(Y^1, Y^2, and Y^3 = 1,4-phenylene groups substituted by Cl, CH_3, OCH_3, and NO_2; X^1 and X^2 = bonds or CONH or SO_2). They have good covering power and good fastness to heat, light, migration, and solvents. Some of them precipitate nitrocellulose. C.O.C.

Reactions and Absorption Spectra of Derivatives of ω-Nitrostyrene. II— Syntheses of Azo Dyes containing a β-Nitrovinyl Group
L. Skulski and T. Urbański
Roczniki Chem., **34** (1960) 431–441 (*English summary*)
Chem. Abs., **54** (25 Nov 1960) 24475

Dyes containing the *p*-(β-nitrovinyl)phenylazo (I) group were made by coupling diazotised 4-(β-nitrovinyl)aniline at pH 8 with the following (position of I group given first): *p*-, phenol (III); *p*-, dimethylaniline (IV); 5-, salicylaldehyde; 5-, salicylic acid; 1-, 2-naphthol (V); 4-, 1-naphthol (VI); 1-, 2-naphthylamine; 4-, 1-naphthylamine. *p*-(β-Nitrovinyl)azobenzene (VII) was prepared from II and nitrosobenzene. III to VII were also made in good yields by reaction of nitromethane with the aldehyde group of the respective azo dyes, derivatives of *p*-aminobenzaldehyde. Purification of all the compounds was difficult. Their absorption spectra are given. C.O.C.

Azo Coupling of Pyrrole and Cyanine Dyes
A. Triebs and R. Zimmer-Galler *Ann.*, **627** (1959) 166–181
Chem. Abs., **54** (25 Nov 1960) 24665

Several dyes were prepared, using *p*-nitroaniline and sulphanilic acid as diazo components, and coupling with, e.g., bis[*N*-ethylbenzothiazole-(2)]trimethincyanine iodide and 2,4-dimethyl-3-carbethoxypyrrole. E.S.

Production of the Dye Direct Black
I. Kurdov, P. Ilinov, and L. Antonov
Tezhka Prom., **5**, No. 8, 34–38 (1956)
Chem. Abs., **54** (25 Nov 1960) 25831

This dye, basically the trisazo compound C.I. 30235, *m*-phenylenediamine ← benzidine → (*acid*) H acid ← (*alk*) aniline, was shown to contain at least 5 other dyes. Purity and quality are influenced by purity of intermediates and by coupling conditions. E.S.

Aromatic Diazo and Azo Compounds
Z. J. Allan and J. Podstata
XXXVI— Coupling with Carbazole, Diphenylamine, and Acetone
Collection Czechoslov. Chem. Communs., **25** (1960) 1324–1336 (*in German*)
Chem. Abs., **54** (25 Nov 1960) 24570

Diazotised 2,4-dinitroaniline coupled with carbazole gives mainly the 2-azo compound, with diphenylamine mainly the 4-azo compound, and with acetone mainly 2,4-dinitrophenylazoacetone, but other compounds are formed by varying conditions of acidity etc. during coupling.

XXXVII— Double Coupling with γ Acid
Ibid., 1337–1343
Chem. Abs., **54** (25 Nov 1960) 24571

The acid-coupled monoazo compound anthranilic acid → γ acid is treated with $NaHSO_3$ and SO_2, which converts the NH_2 group into OH. A second diazo compound can then be coupled with the resulting hydroxymonoazo compound. E.S.

Bis(*p*-Hydroxyphenyl)alkanes as Coupling Components in Cold Dyeing. I— Affinity of Bis(*p*-hydroxyphenyl)alkanes for Cellulosic Fibres
V. N. Klyver and T. S. Meshkova
Tekhnol. tekstil. prom., (3) (1960) 113–117
Chem. Abs., **54** (25 Nov 1960) 25839

At 40°C the affinity for cellulose of bis(*p*-hydroxyphenyl)methane, 1,1-bis(*p*-hydroxyphenyl)ethane, 2,2-bis(*p*-hydroxyphenyl)propane, 2,2-bis(*p*-hydroxyphenyl)butane, and 1,1-bis(*p*-hydroxyphenyl)cyclohexane is very similar to that of derivatives of 2-hydroxynaphthoic acid. These compounds can be used as azoic coupling components. C.O.C.

Azoic Dyes Containing the Morpholine Ring
J. Nishino, A. Nishiura, N. Kuroki, and K. Konishi
Bull. Univ. Osaka Prefect., Ser. A, **7** (1959) 71–77
Chem. Abs., **54** (25 Nov 1960) 25833

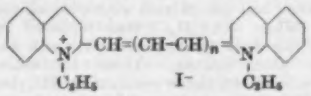
Amines containing a morpholine ring were used as diazo components, and amides from aminophenylmorpholines condensed with 3,2-hydroxynaphthoic acid were used as coupling components, in the preparation of azoic dyes on cotton and Vinylon. Thus 3-chloro-6-morpholinoaniline (I) diazotised and coupled with 3-hydroxy-2-naphthoanilide gives a brown, and I condensed with 3,2-hydroxynaphthoyl chloride gives with diazotised 2,5-dichloroaniline a bright red. E.S.

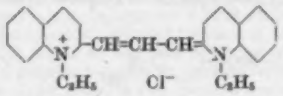
Azoic Dyes Containing the Pyrazolone Ring
J. Nishino, A. Nishiura, N. Kuroki, and K. Konishi
Bull. Univ. Osaka Prefect., Ser. A, **7** (1959) 79–84
Chem. Abs., **54** (25 Nov 1960) 25831–3

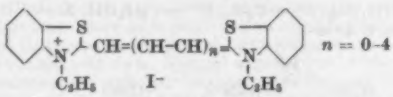
Various aminophenylazo derivatives of pyrazolones were used as diazo components in producing azoic dyes on cotton with arylides of 3,2-hydroxynaphthoic acid. Also, by condensing these aminophenylazo pyrazolones with 3-hydroxy-2-naphthoyl chloride, azoic coupling components were produced. Thus diazotised 4-*p*-aminophenylazo-3-methyl-5-pyrazolone (I) with the anilide of 3,2-hydroxynaphthoic acid gives dark purple, and I condensed with 1 mol. of 3-hydroxy-2-naphthoyl chloride couples with diazotised 2,5-dichloroaniline to give yellow orange. E.S.

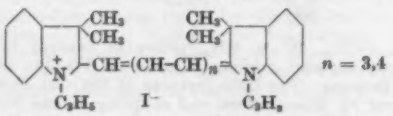
Electron Paramagnetic Resonance Spectra of Cyanine Dyes
Yu. Sh. Moshkovskiĭ *Doklady Akad. Nauk. S.S.S.R.*, **130**, No. 6 (Feb 1960) 1277–1279

The electron paramagnetic resonance spectra of the following dyes were examined—









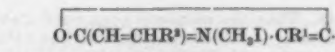
The electron paramagnetic resonance signal was not given by all dyes. The concn. of unpaired electrons is one free electron per 10^4 dye mol. Resonance absorption for a given homologous series of dyes appears with a definite number of conjugations. The appearance of the electron paramagnetic spectrum is due to uncompensated electron magnetic moment, which is attributed to the basic character of the dye molecule. T.Z.W.

Monomethincyanines
N. S. Dokunikhin and Ya. B. Shteĭnberg
Zhur. Vsesoyuz. Khim. Obshchestva im D.I. Mendeleeva, **5** (1960) 239–240
Chem. Abs., **54** (25 Nov 1960) 24795

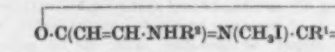
2-Methylbenz[*cd*]indolinium methiodide (I) was prepared by boiling *N*-methyl-2-methylthiobenz[*cd*]indolinium iodide with $CH_3(COOC_2H_5)_2$, $(C_2H_5)_3N$ and abs. C_2H_5OH, cooling, filtering and heating the product with conc. H_2SO_4, evaporating, adding KI, filtering and washing. I was condensed with 2-methyl-substituted bases to give dyes for acrylic fibres. Thus with (base, *N*-substituent on base, λ in mμ), benzothiazole, C_2H_5, 554; quinoline, C_2H_5, 560; benzoxazole, C_2H_5, 497 and 531; 3,3-dimethyl-3*H*-indole, CH_3, 535; and benz[*cd*]indole, CH_3, 645. A.T.P.

Synthesis of Heterocyclic Compounds of Nitrogen. CXXV— Photosensitising Dyes
T. Takahashi and M. Hayami
Yakugaku Zasshi, **80** (1960) 895–901
Chem. Abs., **54** (25 Nov 1960) 24656

d,l-Leucine with acetic anhydride in pyridine gave 3-acetamido-5-methyl-2-hexanone, which on treatment with H_2SO_4 at 0°C or with PCl_5 in benzene gave 2,5-dimethyl-4-isobutyloxazole (A). Similarly, *d,l*-alanine gave 3-acetamido-2-butanone and 2,4,5-trimethyloxazole (B). From A-CH_3I (and B-CH_3I) are obtained styryl-type dyes—



(R^1 = CH_3 or $(CH_3)_2CHCH_2$, R^2 = $X{\cdot}C_6H_4$, X = e.g. *p*-$(CH_3)_2N$, *p*-OH, *m*-NO_2, etc.). Reaction of $A{\cdot}CH_2I$ and $B{\cdot}CH_2I$ with formamidines by fusion on an oilbath gives the aminovinyl dyes—



(R^1 as above, R^2 = 5-bromo-2-pyridyl or $X{\cdot}C_6H_4$, X = e.g. *o*-, *m*-, or *p*-CH_3, *p*-$COOC_2H_5$, etc.). Some antibacterial activities of these dyes are also described. A.T.P.

Investigations in the Anthraquinone Series. XXVIII — Chlorination of Anthraquinone-α-sulphonic Acid with Chlorates

V. V. Kozlov and A. A. Davÿdov

Zhur. obshch. khim., **30** (Oct 1960) 3456–3464

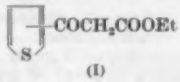
Effect of concn. of $KClO_3$, HCl, H_2SO_4, and a number of chlorides and sulphates on the replacement of the SO_3H group in anthraquinone α-sulphonic acid by Cl is studied in detail. G.J.K.

Derivatives of Heterocyclic Acylacetic Esters. III— Synthesis of α- and β-Thenoylacetic Esters, Arylides, and Azomethin Dyes

N. S. Vul'fson and V. E. Kolchin

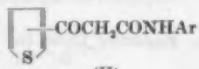
Zhur. obshch. khim., **30** (Oct 1960) 3425–3430

Acylation of the ethoxymagnesium deriv. of acetoacetic ester with α- or β-thenoyl chloride and ammonolysis of the resulting α- and β-thenoylacetoacetic esters yields α- or β-thenoylacetic esters (I)—



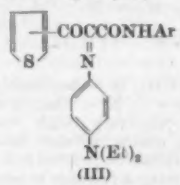
(I)

I with aniline, *o*-, *m*-, *p*-chloroaniline, *o*-, *m*-, *p*-nitroaniline, *o*-, *m*-, *p*-anisidine, and *o*-, *m*-, *p*-aminobenzoic acid gives arylides (II)—



(II)

II with *NN*-diethyl-*p*-phenylenediamine gives, under conditions of oxidative condensation in the presence of AgCl, yellow azomethin dyes (III)—



(III)

The physical constants of II and III are tabulated. G.J.K.

Influence of Soaping on Thioindigoid Dyes

L. M. Golomb and M. G. Romanova

Tekstil. prom., **20** (Jan 1960) 56–58

Soaping of materials dyed with thioindigoid dyes causes a physical condensation of dye molecules and their simultaneous orientation in relation to the axis of the substrate. In many cases this results in a change of hue. Many spectra of soaped dyes show a second definite shift of the absorption towards long wavelengths, which is the property of the *trans*-dye form. T.Z.W.

Continuous Preparation of Chrome Yellows (C.I. 77600)

M. A. Shtern and G. N. Gorelik

Lakokrasochnye Materialy i ikh Primenenie, (2) (1960) 50–55

Chem. Abs., **54** (25 Nov 1960) 25877

Data are given which were obtained from laboratory studies of continuous processes for preparing $Pb(NO_3)_2$ (I) and $Pb(OH)_2{\cdot}Pb(NO_3)(NO_3)$ (II) to replace PbO in preparation of Chrome Yellows of superior properties. I and II were obtained from 3–5 mm Pb pellets and, respectively, 10 and 5% HNO_3 at 90 and 95°C; the processes were made automatic by maintaining the solutions and effluent at pH 2·5 and 5·9–6·7, respectively. II is not a mixture because its composition is unaffected by recrystallisation. C.O.C.

Preparation of Red Molybdate Chromes (C.I. Pigment Red 104)

M. A. Shtern and M. V. Sukhanova

Khim. Prom., (1959) 584–586

Chem. Abs., **54** (25 Nov 1960) 25877

The brightest red molybdate chromes vary in composition from $CrO_4{\cdot}PbMoO_4{\cdot}PbSO_4$ to $27PbCrO_4{\cdot}4PbMoO_4{\cdot}PbSO_4$. The colour is due entirely to $PbCrO_4$, since $PbMoO_4$ and $PbSO_4$ are both white. The colour during formation changes from lemon (rhombic form) to light red and then to dark yellow. The effect of the rate of addition of precipitants to Pb nitrate on the colour of the pigment was studied. The temperature was 20°C, pH 2, and strength of solutions 0·1M. Crystallisation of rhombic $PbCrO_4$ is faster than its recrystallisation into the tetragonal form. As the pH is lowered to 1·8, the red tone deepens. Below pH 1·8 the colour is unstable. 1–2% $Al(OH)_3$ and silicic acid stabilise the pigment. C.O.C.

Manufacturing Problems of Zinc Chromates (C.I. 77955) for Use as Corrosion-inhibiting Pigments

R. Hübner, E. Uhlworm, H. Beandt, and H. Schaller

Plaste u. Kautschuk, **7** (1960) 209–211

Chem. Abs., **54** (25 Nov 1960) 25878

Study of the protection against corrosion offered by 12 types of Zn chromate revealed no correlation between chemical composition and physical behaviour. C.O.C.

PATENTS

Aromatic Perchloryl Compounds— Dye Intermediates

Pennsalt Chemicals Corpn.

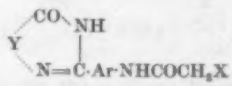
BP 860,309 (U.S.A. 27 Sept 1958)

Perchloryl fluoride, $FClO_3$, reacts with aromatic compounds in presence of a Friedel-Crafts catalyst to yield compounds in which the F atom of the perchloryl fluoride is replaced by an aromatic radical. The resulting primary perchloryl aromatic compounds can be used *inter alia* as intermediates for dyes. C.O.C.

Colour Couplers

Gevaert Photo-Producten *BP* 856,158 (15 Mar 1957)

Compounds of formula—

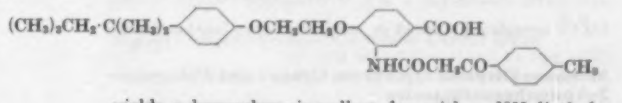


(Y = atoms to complete a 4-quinazolone nucleus; Ar = arylene; X = carboxylic acyl), e.g. 2-(*p*-benzoylacetaminophenyl)-4-quinazolone, are photographic colour couplers having high solubility in developers yet yielding insoluble dyes which do not bleed out. C.O.C.

Yellow Colour Couplers

Gevaert Photo-Producten *BP* 857,028 (20 Mar 1957)

Benzoyl acetanilide derivatives containing a subst. or unsubst. alkoxy group in the *ortho*-position of the anilide group and a –COX (X = OH or NH_2 subst. by Alk or phenyl subst. in turn by a water-solubilising group) are yellow colour couplers of use in negative material, as the dyes they yield absorb in a region where the secondary side-absorption of the magenta is low. Thus—



yields a hypsochromic yellow dye with a *NN*-diethyl-*p*-phenylenediamine developer. C.O.C.

Colour Couplers containing Long-chain Alkylamino-Isophthalic Ester Groups

General Aniline *BP* 857,213 (U.S.A. 31 Oct 1957)

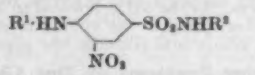
Incorporation of a 5-alkylaminoisophthalic acid (Alk of > 9 C) into colour couplers gives them high solubility in the usual solvents, low m.p. and fastness to diffusion, and renders them readily purifiable by recrystallisation. C.O.C.

Light-sensitive Diazonium Salts
Kalvar Corpn. *USP* 2,923,703 (28 Dec 1954)

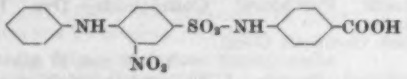
Double salts of 1-dimethylamino-4-naphthalene diazonium halides, prepared e.g. by coupling diazotised sulphanilic acid with *NN*-dimethyl-α-naphthylamine, reducing to give the 4-amino compound and diazotising in presence of fluoroboric acid to give the diazonium fluoroborate, are decomposed by violet or blue light. They can therefore be used for making photographic records by incorporating them in gums, resins, etc. which are altered in appearance by the nitrogen gas evolved when they decompose. E.S.

Nitrodiphenylamine Dyes
Celanese Corpn. of America *USP* 2,921,945 (17 May 1954)

Yellow dyes suitable for cellulose acetate, organic derivatives of cellulose, polyamides and unions thereof, preferably applied at pH 5·5–6·0, have formula—



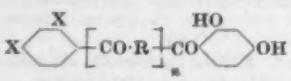
(R^1 and R^2 = subst. or unsubst. Ar, at least one carrying –COOH). They are obtained by known methods and have excellent washing fastness, which may be further improved by aftertreatment with cationic resin-forming materials or metallic compounds. Thus, *p*-aminobenzoic acid is condensed in the usual manner with 4-chloro-3-nitrobenzenesulphonyl chloride, and the product further condensed with aniline to give—



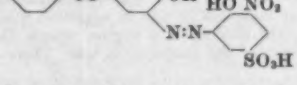
which dyes secondary acetate a fast yellow from a dyebath containing ammonium sulphate. A.T.P.

Metallisable Azo Dyes from 1,3-Dihydroxyphenyl Ketones
FH *BP* 860,279 (Germany 27 Oct 1956)

Mono-, dis-, and poly-azo dyes are formed by coupling suitable diazo compounds with compounds—



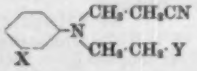
(R = Alk or phenyl; one X = OH, the other X = H or OH; *n* = 0 or 1) made by condensing resorcinol with carboxylic acid chlorides, or with carboxylic acids in presence of BF_3. The diazo components contain OH or COOH in *o*-position to the diazo group, so that the resulting dyes may be metallised in substance or on the fibre. Thus diazotised 2-amino-6-nitrophenol-4-sulphonic acid coupled with 2,2′,4-trihydroxybenzophenone gives the red–brown afterchrome dye—



Its Cr complex prepared in substance dyes leather claret. E.S.

Monoazo Disperse Dyes from Cyano- and Thiocyano-2-Aminobenzothiazoles
ICI *BP* 859,900 (3 Nov 1958)

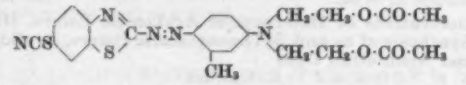
6-Cyano (or thiocyano)-2-aminobenzothiazole is diazotised and coupled with amines—



(Y = CN or O·CO·Alk; X = H, Hal, CH_3, CF_3, etc.) to give disperse oranges and reds of good fastness to light and heat treatments. Thus the monoazo compound 2-amino-6-cyanobenzothiazole → *NN*-bis(β-cyanoethyl)-*m*-toluidine dyes cellulose acetate and Terylene scarlet.

BP 859,899 (3 Nov 1958)

2-Aminobenzothiazoles containing a CN or SCN group (preferably in the 6-position) and optionally other substituents, e.g. Cl, are diazotised and coupled with amines $Ar\text{-}NR^1R^2$ (Ar is of benzene or naphthalene series; R^1 = H, aralkyl or subst. or unsubst. Alk; R^2 = subst. or unsubst. Alk) to give orange to blue disperse dyes, the dyes of *BP* 859,900 being excluded. When R^1 or R^2 is β-cyanoethyl, or when either is or both are β-carbethoxyethyl or γ-chloro-β-hydroxypropyl groups, dyes particularly suitable for Terylene are obtained, whereas if R^1 and R^2 represent β-carbo- lower alkoxy-ethyl or β-acyloxy-ethyl (the acyl group being derived from a lower alkyl carboxylic acid, e.g. acetic acid), the dyes are particularly suitable for cellulose acetate. Thus the monoazo compound 2-amino-6-thiocyanobenzothiazole → *NN*-bis(β-acetoxyethyl)-*m*-toluidine—



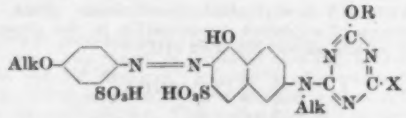
gives bluish reds of good fastness to dry heat on cellulose acetate, nylon, and Terylene. E.S.

Metal(Cobalt and Chromium)-complex Monoazo Compounds having Cyano Groups— Dyes for Wool, etc.
Ciba *BP* 859,275 (Switzerland 21 Jan 1957)

Derivatives of *o*-aminophenol, *o*-alkoxyaniline, or anthranilic acid containing a CN group, are diazotised and coupled with *o*-coupling hydroxy or amino compounds to give monoazo compounds whose 1:2 complexes with Co or Cr dye wool, etc. from neutral or weakly acid baths. Thus the Cr complex of the monoazo compound 2-amino-4-cyanophenol → 2-naphthol-6-sulphonamide is violet. E.S.

Scarlet Monoazo Reactive Dyes for Cellulose
ICI *BP* 859,989 (26 Mar 1958)

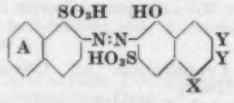
Reactive dyes—



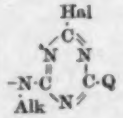
(Alk = preferably CH_3; R = preferably CH_3, but may be Ar or Alk of up to 4 C; X = Hal, preferably Cl) can be incorporated in relatively high concentrations into thickened printing pastes without causing gelatinisation, so that deep scarlets can be printed free from specks. Thus 2,4-dichloro-6-methoxy-*s*-triazine is condensed with 1 mol. of the aminomonoazo compound obtained by coupling diazotised 4-aminoanisole-3-sulphonic acid with *N*-methyl-*N*-acetyl-J acid and hydrolysing to remove the acetyl group. The product (R and both Alk groups = CH_3; X = Cl) gives scarlets on cellulose when applied in presence of an acid-binding agent. E.S.

Monoazo Reactive Dyes for Cellulose
ICI *BP* 859,990 (26 Mar 1961)

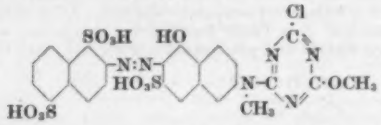
The title dyes have formula—



(ring A may contain further SO_3H groups; X = H, Hal, or SO_3H; one Y = H or SO_3H; the other Y is a group—



where Q = radical of < 14 C bound to the triazine nucleus through C, N, O or S) and give oranges and reds of good fastness to washing in presence of hypochlorites. Thus the acetyl group of the monoazo compound 2-naphthylamine-1,5-disulphonic acid → *N*-acetyl-*N*-methyl-J acid is removed by hydrolysis with boiling aq. NaOH and the product is condensed with 1 mol. of 2,4-dichloro-6-methoxy-*s*-triazine to give—

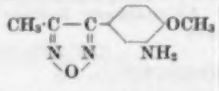


which gives orange on cotton when dyed or printed in presence of an acid-binding agent. E.S.

Oxdiazole (Furazan) Derivative—Diazo Component for Azoic Dyes and Pigments

FH *BP* 859,444 (Germany 6 June 1956)

3 - (3′ - Amino - 4′ - methoxyphenyl) - 4 - methyl - 1,2,5-oxdiazole—

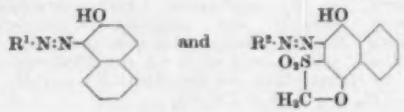


diazotised and coupled, in substance or on the fibre, with arylides of 3-hydroxy-2-naphthoic acid (or of its 7-halogeno derivatives) gives reds of good fastness. Thus on cotton impregnated with the 5-chloro-2,4-dimethoxyanilide of 3-hydroxy-2-naphthoic acid it gives bordeaux. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, etc.

BASF *BP* 858,842 (Germany 23 Dec 1957)

Complexes from 1 atom of Co or Cr and 2 mol. of different monoazo compounds—

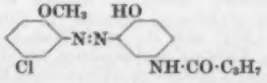


(R^1, R^2 = residues of *o*-aminophenols, at least one of which contains an alkylsulphone or sulphonamide group, but both are free from SO_3H and COOH groups) dye wool from weakly acid baths, the Cr complexes being mainly blues and the Co complexes bordeaux. Thus a mixture of 1 mol. of each of the monoazo compounds 2-aminophenol-4-sulphonamide → β-naphthol and 2-aminophenol-5-sulphonamide → 4′-hydroxynaphtho(2′,1′:4,5)-oxathiol-*s*-dioxide in aq. NaOH is stirred with $K_2Cr_2O_7$ and glucose at 90–95°C to give a mixed complex which dyes wool navy blue. E.S.

Metal-complex Monoazo Dyes

BASF *BP* 858,590 (Germany 18 Apr 1957)

2-Alkoxyanilines, which may carry substituents, e.g. Alk, OAlk, $SO_2{\cdot}NH_2$, in the 5-position are diazotised and coupled with *o*-coupling 2- or 4-*N*-acylaminophenols to give metallisable monoazo compounds. The 1:2 Cr or Co complexes, or mixed complexes with other metallisable dyes, dye wool, nylon, etc. Thus diazotised 2-amino-4-chloroanisidine coupled with 4-*N*-butyrylaminophenol gives—

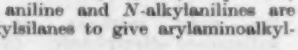


whose Co complex dyes wool bordeaux from a weakly acid bath. E.S.

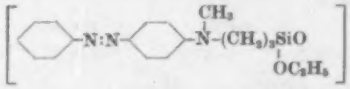
Siloxane Azo Dyes

Union Carbide Corpn. *USP* 2,925,313 (29 Apr 1957)

Arylamines such as aniline and *N*-alkylanilines are condensed with haloalkylsilanes to give arylaminoalkylsilanes—



(R = H or Alk; V = Alk or Ar; Y = OAlk or OAr; *a* = integer, preferably 3–5; *n* = 0, 1 or 2) which couple with diazo compounds to give azo dyes, the type depending largely on the type of diazo compound used. Monomeric silane dyes are formed under anhydrous conditions, but in presence of water polymeric siloxane dyes result. Thus, diazotised aniline coupled in presence of water with *N*-methyl-*N*-(γ-triethoxysilylpropyl)aniline gives the red polysiloxane dye containing the unit—

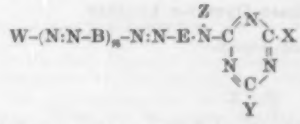


E.S.

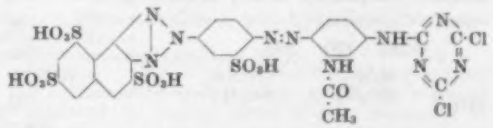
Mono- and Dis-azo Compounds Containing Triazole Rings—Reactive Dyes for Cellulose

ICI *BP* 859,198 (7 Nov 1958)

The title dyes have formula—



(W = R·A– or P·Q–; A, B = divalent radicals of benzene or naphthalene series; E = residue of a coupling component; R, Q = benzotriazolyl or naphthatriazolyl radicals; P = Ar of benzene or naphthalene series attached to Q through an N of the triazolyl ring; X = Hal; Y = Hal, Alk, Ar, OAlk, NH_2, etc.; Z = H or Alk; *n* = 0 or 1) and contain at least 2 SO_3H groups. The triazole rings are introduced by treating a suitable intermediate containing an *o*-aminoazo group with ammoniacal $CuSO_4$. If a chelatable grouping is present, metal complexes may be formed, especially with Cu. Thus the monoazo compound 4-nitroaniline-3-sulphonic acid → 1-naphthylamine-3,6,8-trisulphonic acid is treated with ammoniacal $CuSO_4$ to give the triazole, the NO_2 group is reduced to NH_2, and the product is then diazotised and coupled with *m*-aminoacetanilide. Condensation with cyanuric chloride, followed by isolation in presence of buffers giving pH 6·8, yields—



which dyes cellulose reddish yellow when applied in conjunction with an acid-binding agent. Over 60 examples of dyes are given, their hues ranging from yellow to blue. E.S.

Mono- and Dis-azo Dyes from 3-Methyl-1-Phenyl-4-Pyrazolone

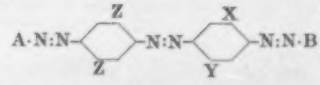
BASF *BP* 858,562 (Germany 22 Feb 1957)

3-Methyl-1-phenyl-4-pyrazolone (I) and its derivatives substituted in the phenyl nucleus by Hal, SO_3H, $SO_2{\cdot}NH_2$, etc. (*BP* 785,185), couple with diazo and tetrazo compounds to give mono- and dis-azo dyes or pigments, deeper in hue than the isomers from the corresponding 5-pyrazolones. Thus *p*-chloroaniline → I is a red-brown pigment; 5-nitro-orthanilic acid → I is a black acid dye, and R acid ← benzidine → I is a red-brown direct dye. E.S.

Metal(Copper)-complex Trisazo Direct Dyes

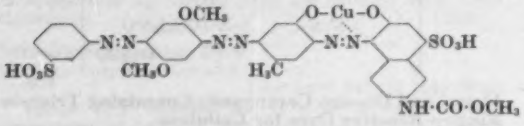
Gy *BP* 858,481 (Switzerland 12 Apr 1957)

Copper complexes derived from trisazo compounds—



(A = residue of benzene- or naphthalene-sulphonic or carboxylic acid, but free from complexing groups; B = residue of 1,3(or 2,4)-naphtholsulphonic acid having a 6(or 7)-acylamino group; X = OAlk, hydroxyalkoxy or carbomethoxy group capable of complex formation; Y = H, Hal, Alk, OAlk or hydroxy-alkoxy; Z = Alk, OAlk or

hydroxyalkoxy, and one of them may be H or Hal) dye cotton green or grey, and are faster and/or of better dyeing properties than similar dyes in which one or both of the middle components is of the naphthalene series. Thus the aminomonoazo compound metanilic acid → 2,5-dimethoxyaniline is diazotised and coupled with *p*-cresidine, and the aminodisazo compound so formed is then diazotised and coupled with *N*-carbomethoxy-γ acid. Heating with ammoniacal $CuSO_4$ in presence of diethylamine then gives the complex—

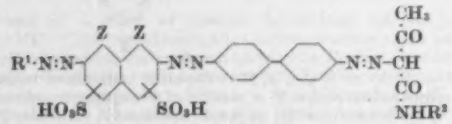


which dyes cellulose green–grey, little affected by anti-crease processing. E.S.

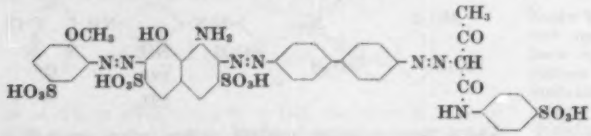
Green Trisazo Dyes for Leather

FH *BP* 858,556 (Germany 21 Dec 1956)

Trisazo compounds—



(R^1 = Ar of benzene or naphthalene series; R^2 = residue of an arylsulphonic acid; one Z = OH, the other = NH_2; the SO_3H groups are in 3,6- or 4,6-position relative to NH_2 = 1; the benzidine residue may carry substituents) dye leather well-penetrated bright greens, and are more soluble than the similar dyes of *BP* 526,015 (J.S.D.C., **57** (1941) 32), in which non-sulphonated acetoacetarylides are used as end components, so that they can be applied by brush-dyeing from a cold bath. Thus benzidine is tetrazotised and acid-coupled with 1 mol. of H acid, and this diazomonoazo compound is then coupled with a solution of diazotised 2-aminoanisole-4-sulphonic acid in presence of soda ash. Finally a soln. in aq. NaOH of *N*-acetoacetylsulphanilic acid is added to give—

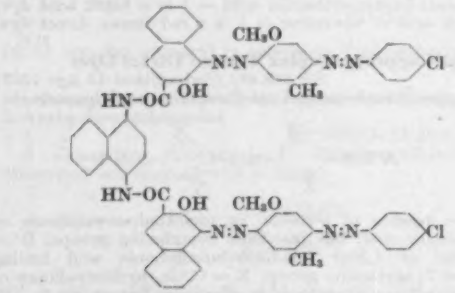


E.S.

Tetrakisazo Pigments

Ciba *BP* 860,278 (Switzerland 23 Oct 1956)

The COOH groups of disazo dyes derived from 3-hydroxy-2-naphthoic acid are converted into COCl groups, which are then condensed with aromatic diamines to give tetrakisazo pigments. Thus the disazo compound 4 - amino - 2 - methyl - 5 - methoxy - 4′ - chloroazobenzene → 3-hydroxy-2-naphthoic acid is treated with $SOCl_2$ in *o*-dichlorobenzene, and 2 mol. of the acid chloride so formed are condensed with 1 mol. of 1,4-diaminonaphthalene to give—

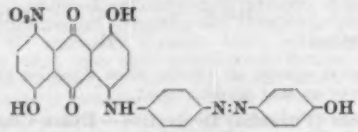


which colours polyvinyl chloride reddish blue. E.S.

Green Dyes for Polyesters

Crompton & Knowles *USP* 2,929,810 (23 June 1958)

4,5-Dinitrochrysazin (I) and 4,8-dinitroanthrarufin (II) are condensed with mildly basic azoanilines free from bulky substituents *ortho* to the $-NH_2$ group to give green disperse dyes for hydrophobic fibres. Thus, II (16·5), 4-(*p*-aminophenylazo) phenol (24), nitrobenzene (2·6) and 2-butoxyethanol (56) are heated 18 h at 150–155°C. The hot liquor is poured on to 1% aqueous NaOH (700) and 30% HCl added to precipitate the dye. After washing by decantation, it is twice purified by extraction with 1% aqueous NaOH and precipitation with 30% HCl to give—

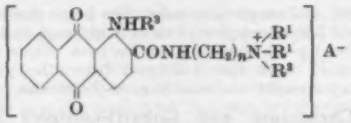


which dyes polyester fibres clear green of outstanding fastness to light, washing, and sublimation. A.T.P.

(1 - Alkylamino - 2 - anthraquinonylcarbonylamido) Alkyl Trialkyl Ammonium Salts—Disperse Dyes for Acrylic Fibres

A. W. Joyce *USP* 2,924,609 (25 Sept 1957)

Cationic anthraquinones—

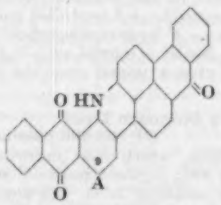


(R^1 = lower Alk, R^2 = lower Alk, aralkyl or hydroxyalkyl, R^3 = Alk or cyclohexyl, n = 2–4, A = anion) are water-soluble dyes having excellent affinity for polyacrylonitrile fibres of high acrylonitrile content. They are obtained, e.g. by condensing 1-nitroanthraquinone-2-carbonyl chloride and dimethylaminopropylamine, replacing $-NO_2$ by heating with ethylamine in toluene, and finally quaternising with, e.g. dimethyl sulphate, to give the strong bluish red dye where R^3 = C_2H_5, R^1 = R^2 = CH_3, n = 3, A = $CH_3SO_4^-$. A.T.P.

Anthranaphthacridone Vat Dyes

American Cyanamid Co. *BP* 860,384 (17 June 1957)

Brown vat dyes of excellent affinity and light fastness are—

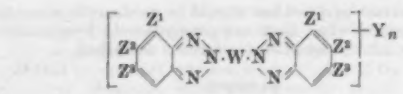


(A = 1-nitro(or amino)-anthraquinone-2-carboxamido radical, optionally substituted) and their mono- and di-halogenated derivatives, or mixtures thereof. Thus, 9-aminonaphthacridinetrione (116) is heated at 140°C with nitrobenzene (2000) to remove water. 1-Nitroanthraquinone-2-carbonyl chloride (I) (104) is added in portions at 150°C and the mass heated at 200–205°C. After cooling, filtering, and washing, the chocolate-brown vat dye contains 2% chlorine, chlorination having occurred simultaneously with condensation, probably because of Cl_2 liberation by oxidation of I by nitrobenzene. If 1-methylnaphthalene is used as solvent, the product contains no chlorine. A.T.P.

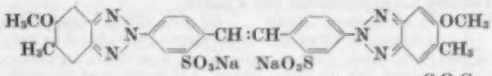
Bis - *pp*(*p* - methoxy - *m* - methylbenzotriazolyl) stilbene-*oo*′-disulphonic Acid Disodium Salt—Fluorescent Brightening Agent

General Aniline *USP* 2,927,866 (15 Nov 1954)

Compounds of formula—



(W = divalent residue of a styrene, fluorene, diphenyl, dibenzofuran, carbazole, dibenzothiophen-5,5-dioxide, naphthalene, dinaphthyl, acridine, pyrene compound or an alkyl, alkoxy, or halogenated derivative thereof; Z^1, Z^2 and Z^3 = H, Alk, alkoxy, halogen, acylamino, alkyl sulphamido, arylsulphamido, *NN*-dialkylamino; y = water-solubilising group; $n < 5$) are fluorescent brightening agents particularly useful for cellulosic fibres. They are much faster to hypochlorite than known bis[(substituted 2,4 - diamino) - 1,3,5 - triazinyl(6)]4,4′ - diaminostilbene-2,2′-disulphonic acids, aroylamides of 4,4′-diaminostilbene-2,2′-disulphonic acid or their salts. They are substantive to cellulose from soap or detergent solutions and their fluorescence has no undesirable greenish cast. The preferred compound is—

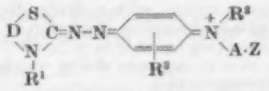


C.O.C.

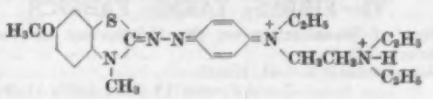
Cationic Diazacyanine Dyes

BASF *BP* 860,584 (Germany 24 Oct 1957)

The above are—



(D = atoms to complete a thiazole or benzothiazole ring; R^1 = aliphatic of 1–5 C; R^2 = H, aliphatic or aromatic; R^3 = H or Alk of 1–4 C; A = divalent aliphatic, aromatic or aliphatic–aromatic radical which may contain hetero atoms; Z = amino group). They are obtained by (*a*) treating hydrazones of thiazolones-(2) or benzothiazolones-(2) alkylated on ring N and aromatic amines with oxidising agents, or (*b*) diazotising 2-aminothiazoles unsubstituted on ring N, coupling to aromatic amines and alkylating on the thiazole N. They are dyes for tannic-acid-mordanted cotton, cellulose acetate, polyamides, and polyesters, and colour polymers or copolymers of acrylonitrile or dicyanoethylene in fast shades not previously attainable. Thus, a very fast pure greenish blue for polyacrylonitrile is—

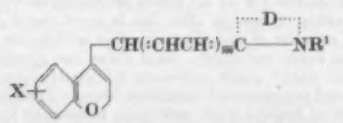


(See also *BP* 786,929 and *BP* 787,369, J.S.D.C., **74** (1958) 97, 98.) A.T.P.

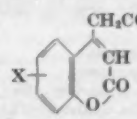
Methin and Polymethin Dyes

Ilford *BP* 856,068 (29 April 1958)

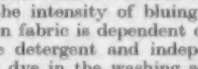
Dyes of formula—



(X = H, one or more substituents, or a fused benz or subst. benz group; R^1 = subst. or unsubst. Alk; D = atoms to complete a 5-membered nucleus; m = 0 or 1), e.g. the yellow 7-hydroxy-4-[(3-methyl-2-benzothiazolinylidene)methyl]-coumarin, have photosensitising properties. They are obtained by condensing a compound of formula—



with one of formula—



(Q = thioether (–SR) or thioethervinyl (–CH:CHSR); R = Alk or aralkyl or acetanilidovinyl; Y = acid radical). C.O.C.

Polar Brilliant Blue GAW (see C.I. Acid Blue 127) as Bluing Agent in Laundry Detergents

Thomas Hedley & Co. *BP* 850,531 (U.S.A. 1 Oct 1956)
Procter & Gamble Co. *USP* 2,930,760 (1 Oct 1956)

Polar Brilliant Blue GAW co-acts with alkaline detergents so that the intensity of bluing in any given washing on any given fabric is dependent on the ratio of active dye to active detergent and independent of the concentration of the dye in the washing solution within a wide range of washing concentrations. Because of this, detergents containing this dye are completely non-staining. This property is apparently unique to this dye. C.O.C.

[*Abstractor's Note:* This abstract replaces that headed "C.I. 61135 as Bluing Agent in Laundry Detergents" on p. 36 of the January *Journal*. That abstract was based on the statement made in these patents that Polar Brilliant Blue GAW has the constitution C.I. 61135. The *Colour Index* Editorial Panel has, however, been informed by the makers of the dye, Messrs. J. R. Geigy S.A., Basel, that this statement is incorrect and that the dye has in fact a constitution somewhat different from that of C.I. 61135.]

Metal Tannates for Pigmenting Film-forming Compositions

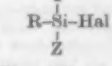
Forestal Land, Timber & Railways Co. and Research Assocn. of British Paint, Colour & Varnish Manufacturers *BP* 860,277 (28 Aug 1957)

Tannin extract in water or other polar solvent is treated with a compound of a polyvalent metal, e.g. Pb acetate or PbO. The product is then incorporated with an oily or resinous medium capable of forming adherent, water-repellent films, e.g. in latex or boiled linseed oil. Usually one atom of metal per catechol or pyrogallol grouping present in the tannin is used. The resultant films have marked anticorrosive properties, a mimosa–lead chelate having the same protective properties as Red Lead (C.I. Pigment Red 105). C.O.C.

Siliceous Pigments Coated with Unsaturated Organohalogenosilanes

Columbia-Southern Chemical Corpn. *BP* 858,555 (U.S.A. 24 Dec 1956)

The rubber-reinforcing properties of siliceous pigments are enhanced by coating them with a compound of formula—



(R = cyclopentenyl; Y and Z = same or different H, Hal or cyclopentenyl), particularly cyclopentenyltrichlorosilane. C.O.C.

Rutile Titanium Dioxide (C.I. Pigment White 6) for Pigmenting Laminates containing Aminoplast Resins

National Lead Co. *BP* 859,729 (U.S.A. 15 Jan 1958)

Rutile TiO_2 coated with hydrous oxides of Al and Si and then calcined, when used in laminates containing melamine- or urea–formaldehyde resins, yields a product which is resistant to discoloration by sunlight. C.O.C.

Mass Coloration of Polyamides (VI p. 182)
Measuring Complexing Affinities of Aromatic Tanning Agents and Dyes for Chromium (III) Salts (XIV p. 190)
Analytical Study of the Natural Red Food Dye Alkannin (C.I. 75530) (XIV p. 190)

V— PAINTS; ENAMELS; INKS

Physical Chemistry of Interfaces Related to Coatings

W. C. Prentiss *Off. Dig. Fed. Soc. Paint Technol.*, **32** (Dec 1960) 1611–1617

Review of material drawn from an earlier symposium, illustrating the relationship between colloids, interfaces, and coatings. J.W.D.

Utilisation of Mica (C.I. Pigment White 20) in Paints
V. L. Levinav and M. S. Leĭzerzon
Trudy Vsesoyuz. Nauch.-Issledovatel. Inst. Asbestos, Slyudy, Asbestotsement. Izdeliĭ i Proekt. Stroitel'stva Predpriyatiĭ Slyud. Prom., (9) (1958) 163–174
Chem. Abs., **54** (25 Nov 1960) 25874

Study of possible use of two forms of ground Mica, Muscovite and Phlogopite, as fillers for oil paints. Their physicochemical properties and their mesh composition, characterising the grinding difficulties, are given. Both forms had low *n* values (1·5) and showed no spreading capacity even when mixed with TiO_2 (C.I. Pigment White 6), and because of this the trials were made by using Mica as filler mostly in Ti and Zn White and partly with Ochre (C.I. 77491 and 77492) and Yellow Iron Oxides (C.I. 77491 and 77492). The addition somewhat increased the oil capacity of the Ochre and Iron Oxide, but made grinding difficult; the Mica did not affect the elasticity, stability to bend and impact-resistance during ageing. Addition of Muscovite to Zinc White (C.I. Pigment White 4) (1:1) and of phlogopite (1:3) improved the anti-corrosion properties of the pigment. Similar results were obtained by adding them to a mixture of TiO_2 and ZnO. Addition of Mica to TiO_2, Ochre and Yellow Iron Oxides did not improve their protective properties. Addition of Mica to oil paints based on a mixture of Zn and Ti Whites improved their weathering properties remarkably, but this was not the case with Ochre and Yellow Iron Oxides. C.O.C.

Dispersing Pigments in Alkyd Media
J. Beikmanis and L. Kovacs
Australian Paint J., **5** (3) (1960) 11–13
Chem. Abs., **54** (25 Nov 1960) 25878

A 61% linseed, pentaerythritol alkyd having 50% excess hydroxyl is an excellent grinding medium for many pigments. C.O.C.

Effect of Pigment Concentration on Protective Properties of Pigmented Coatings. I—Effect of Pigment Concentration on Protective Properties of Drying Oils
N. Mjagkovs and L. Liepina
Latvijas PSR Zinātnu Akad. Vēstis, (4) (1960) 109–116 (*in Russian*)
Chem. Abs., **54** (25 Nov 1960) 25881

Drying oil (unspecified) was pigmented with ZnO, Fe_2O_3, TiO_2, and Pb_3O_4 (C.I. 77947, 77491, 77891, and 77578). The resulting paints were brushed on and the protective properties of the dried film determined by electrolysis in presence of aq. 3% KCl. The results showed that the protective properties of the coatings were dependent to a high degree on the kind and concentration of the pigment. The different behaviour of oil-based coatings at the anode and cathode is explained on the basis of electrokinetic properties, and the poor chemical resistance to alkalis. Graphs show the effect of time and pigment concentration on the properties of the coatings. C.O.C.

Effect of Pigment Particle Shape and Size on Moisture Permeability of Organic Coatings
M. Kronstein, H. Abramski, J. Rivera, and F. Weber
Australian Paint J., **5** (2) (1960) 13–21
Chem. Abs., **54** (25 Nov 1960) 25877

Blistering on wood, vapour losses through free films, and light transmission were tested for coatings based on a variety of latexes and alkyds pigmented with TiO_2 (C.I. Pigment White 6), $MgSiO_3$ (C.I. Pigment White 26), and Mica (C.I. Pigment White 20) of various particle sizes. Use of larger, plate-like Mica particles in the TiO_2 improved moisture resistance. A mixture of large and small particles was best, while very small particles decreased the sealing effect. C.O.C.

Improved Printing Inks—II
B. I. Beregin. *Poligraf. Proizvodstvo*, (6) (1955) 22–25
Referat. Zhur., Khim., (1956) Abstr. No. 70003
Chem. Abs., **54** (25 Nov 1960) 25878

A discussion of the quality and spectrophotometric characteristics of some pigments and lakes made for printing inks. Use of Glyptal and Pentaphtal (from pentaerythritol and phthalic anhydride) resins modified by dry oil considerably improves the quality of printing inks. To obtain best brightness and fineness of pigments, they should not be dried but should be used as the aqueous pastes obtained when they are precipitated. Preparation of printing ink by use of such pastes is described. C.O.C.

PATENTS

Flatted and Semi-gloss Coating Compositions
Monsanto Chemical Co. *USP* 2,927,863 (15 Aug 1955)

Finely powdered silica (C.I. Pigment White 27) is added to a lacquer or paint together with a non-ionic surface-active condensate of 7–20 mol. ethylene oxide and 1 mol. of an aliphatic alcohol of 8–16 C. Sufficient silica is used to cause appreciable decrease in gloss of coatings formed from the lacquer and sufficient condensate to prevent hard caking of the silica. C.O.C.

Aqueous Emulsion Paints
Shawinigan Chemicals *BP* 856,527 (Canada 28 Dec 1957)

Describes production of a paint of pigment volume composition 8–80% by polymerising or copolymerising suitable monomers in presence of the usual paint ingredients, including the pigment dispersion. C.O.C.

Tinting Compositions for Paints
Jensen & Nicholson Group *BP* 861,223 (17 Sept 1958)

A mixture of a water-insoluble pigment, a glycol or poly-oxyalkylene diol, and an anionic or non-ionic surfactant can be used for tinting either water-thinned emulsion paints or oil, oleoresinous, or similar paints which are thinned with organic solvents. C.O.C.

Tinting Powdered Cold Water Paints
Welco Manufacturing Co. *USP* 2,927,862 (20 June 1955)

The pigment is mixed with a finely divided granular material having a solid wetting agent of m.p. $> 20°C$ coated on it or with a wetting agent of m.p. $> 20°C$. The mixture is then made into an aqueous slurry which, when added to paint, uniformly tints it. C.O.C.

Paints Based on Portland Cement
Associated Portland Cement Manufacturers
BP 860,069 (10 Oct 1956)

Dry cement base paints are rendered readily wettable and dispersible in water by addition to the dry powder of an aliphatic primary monoamine of 6–10 C, e.g. Armeen 8 (a mixture of C_8–C_{10} amines). C.O.C.

Emulsifying Agents for Resins (III p. 172)
Synthesis of Azo Paints for Car Lacquers (IV p. 173)
Metal Tannates for Pigmenting Film-forming Compositions (IV p. 179)
Curing of Polyepoxides (XIII p. 189)

VI—FIBRES; YARNS; FABRICS

Effect of Weathering on the Properties of Textile Fibres—III
J. Lünenschloss and H. Kurth
Textil Praxis, **15** (Nov 1960) 1146–1150

All the more important fibres (except protein, polyvinyl alcohol, and polyolefin fibres) were examined. Chemical changes brought about by weathering of up to 12 months' duration were followed by estimating the average degree of polymerisation (DP), using the method of Heermann and Agster. The results are presented graphically, and it is evident that the DP of all fibres decreases as the duration of weathering increases. The fall is most rapid during the first 2–4 months of weathering, after which the DP tends to remain fairly constant. As a class, the synthetic fibres are considerably more resistant to weathering than the natural and regenerated cellulosic fibres. Tensile strength, extension at break, work modulus, resistance to abrasion, and DP value after 3, 6, 9, and 12 months of weathering of all fibres are presented in tabular form. The authors conclude by describing apparatus used for artificial weathering. Basically it consists of an enclosed carbon arc. The test samples are mounted on a cylindrical cage rotating at 3 rev/h. During each revolution the patterns are sprayed with water for 1 min. The temperature and relative humidity, measured in the centre of the apparatus, are 32°C and 45% during spraying, and 50°C and 10% in absence of spraying. L.A.T.

Cyanoethylation of Cotton
N. M. Bikales and L. Rapaport
Text. Manuf., **86** (Dec 1960) 500–503

Early processes for the cyanoethylation of cotton with

acrylonitrile resulted in large losses of the latter in side-reactions, and present methods achieve only 50% utilisation of the acrylonitrile. A detailed study of the optimum conditions for cyanoethylation is reported, but the cellulose derivatives obtained were subsequently found to have suffered hydrolysis to such an extent as to be devoid of the desirable rotproofness associated with cyanoethylation. In the reaction of cotton with acrylonitrile in presence of aqueous sodium hydroxide, the degree of cyanoethylation, as measured by the nitrogen content of the product, is proportional to the acrylonitrile concentration, and reaches a maximum with a concentration of 13% sodium hydroxide in the aqueous phase with the minimum quantity of aqueous phase necessary to wet-out the cotton. A reaction time of 2 h at 15–20°C is optimum, and addition of sodium chloride leads to higher nitrogen content in the product. Under the optimum conditions described, about half of the acrylonitrile present was destroyed in side reactions and the cellulose derivatives obtained in this one-stage process showed appreciable swelling in water and lower tensile strength and higher elongation compared with the products of the usual two-stage process in which treatment with alkali precedes reaction with acrylonitrile. The products were also virtually devoid of rot-resistant properties, even though the nitrogen contents attained (5·5%) appreciably exceeded those necessary to confer rot-resistance (3·0%) in the two-stage process. The difference in behaviour is attributed to hydrolysis of some of the nitrile groups of the cyanoethylated cellulose to carboxyl groups in the one-stage process. E.C.

X-ray Crystallinity Estimations of Randomly and Longitudinally Orientated Fibres of Regenerated Cellulose

M. Ruck-Florjancic and H. Ruck

Svensk Papperstidning, **63** (15 Dec 1960) 845–853 (*in English*)

Degree of crystallisation (D.C.) is unaffected by the method of spinning and by the concn. of cellulose in the viscose soln., but increases on prolongation of regeneration. Coagulation produces self-orientation of micelles, but filaments spun under low pressure are randomly orientated. Moderate stretch of 25%, applied during regeneration, results in a sudden increase in D.C., but further elongation has no significant effect. 30 References. R.A.

The Polynosic Fibre

Text. Rec., **78** (Feb 1961) 50–52

The term "polynosic" was coined some years ago by one firm to describe viscose rayons of a special type characterised by a wet modulus of the same magnitude as that of cotton; it has not been adopted as a generic name. The article describes successive modifications to the viscose process that have led to fibres of greater strength, i.e. addition of zinc salts (1912), quaternary ammonium compounds (1950), and other additives. Typical physical properties of polynosic fibres are tabulated and it is concluded that they are due to a molecular chain about twice as long as that of a conventional rayon, together with a unique molecular structure. M.T.

Relationship Between Cross-sectional Staining and Degree of Swelling in Cellulosic Fibres

K. Katō

J. Soc. Textile Cellulose Ind. Japan, **16** (Oct 1960) 831–838

Several swelling treatments of cellulose rayons were investigated and a good correspondence established between decrease in degree of swelling and the transition of cross-sectional stainability from core-stain to skin-stain. It was concluded that swelling-determining regions are probably essentially those determining absorption and retention of dye molecules, i.e. least amorphous, and that the degree of moisture sorption is possibly dependent on the whole regions of amorphous cellulose. A correlation was established between degrees of sorption and of swelling in cellulosic fibres grouped according to their cross-sectional staining behaviour. A.T.P.

Further Development of Synthetic Fibres Based on Cellulose. Processing of the Polynosic Fibre Z.54

L. Martin *Chemiefasern*, No. 1 (Jan 1961) 48–50

The properties of the synthetic fibre Z.54, and their influence on processing, are discussed. They are such that in many cases the machines used in the processing of cotton can be used for this fibre. Desizing, mercerisation, and bleaching of Z.54 and Z.54–cotton mixtures are reviewed. In its dyeing properties, Z.54 shows similarity to other cellulosic fibres, good affinity being exhibited by, for example, normal direct, vat, solubilised vat, reactive, and sulphur dyes. The fastness of dyeings to wet treatment and to light is reported to be as good as that of the corresponding dyeings on cotton. Finishing, particularly the application of resins, is also considered. Resins of the urea type have been applied, Z.54 treated in this way exhibiting good crease resistance, tearing strength, etc. The use of Z.54 in admixture with cotton is reported to result in better mechanical properties and softer handle than are obtained with resin-treated cotton. I.G.L.

Action of Oxidising Agents on Cellulose and Hydrocellulose

W. B. Achwal and G. M. Nabar

Text. Research J., **30** (Nov 1960) 872–881

Hydrolysis with dil. H_2SO_4 decreases susceptibility to oxidation, moisture regain and DP, but the density progressively increases and becomes constant at about the same time as the other factors do. Oxidation of cellulose and of hydrocellulose follow the same mechanism. C.O.C.

Studies on the Fibrous Acetylation of Cotton I— Acetylation of Cotton Fabric by the Liquid Phase Method

W. Tsuji, R. Kitamaru, and Y. Sakaguchi

J. Soc. Textile Cellulose Ind. Japan, **16** (Dec 1960) 1020–1026

Cotton fabrics have been acetylated to various degrees of substitution by the liquid phase method previously reported by Sakurada and Sakaguchi. The tensile properties before and after acetylation have been studied, and it is concluded that, under the right conditions, cotton fabric can be acetylated by this method to a high degree of substitution without any decrease in tensile strength taking place. The preparation and stability of the acetylation baths, and the use of catalysts and diluents are discussed.

II— Acetylation of Cotton Fabric by the Semi-liquid Phase Method (Padding Method)

W. Tsuji, R. Kitamaru, Y. Sakaguchi, and F. Kimura

Ibid., 1026–1031

A method of acetylation is described in which the cotton fabric, after a pretreatment with acetic acid, is immersed in an acetylation bath containing acetic anhydride and a catalyst, squeezed to 60–70% pick-up, and finally heated in a closed vessel. Almost all the acetic anhydride taken up reacts, and the cotton fabric is acetylated to ca. 30 mol. % without any lowering in tensile strength. Because of the short period of immersion and reaction, the method is ideally suited for application as a continuous acetylation process. I.G.L.

Absorption of Saturated Azobenzene Vapour by Cellulose Acetate

F. Jones *J.S.D.C.*, **77** (Feb 1961) 57–62

Action of Dimethyl Sulphoxide on Wool

N. H. Koenig and R. A. O'Connell

Text. Research J., **30** (Sept 1960) 712–714

Dimethyl sulphoxide causes supercontraction of wool (28% decrease in length when treated for 1 h at 105°C) probably because of physical rearrangement of the polypeptide chain. Treatment for > 30 min at 105°C seriously diminishes the mechanical strength of wool and its resistance to acids and alkalis. C.O.C.

Studies on Cystine in Wool. VI— Relationship between Thiol Groups and Supercontraction

Y. Nakamura, Y. Nemoto, and M. Oku

J. Soc. Textile Cellulose Ind. Japan, **16** (Oct 1960) 852–858

Studies of the relation between the thiol group and supercontraction of wool in phenol solution indicated that (i) supercontraction depends more on the total thiol group content than on cross-linkages between keratin molecules, (ii) increase of supercontraction found in alkaline phenol solution could be caused by increase in thiol groups formed by hydrolysis of disulphide bonds, (iii) wools which are less damaged (low alkali solubility) do not always have better shrink-resisting properties, and *vice versa*. The best improvement in both properties is obtained by one-process oxidation of thiol groups and formalisation, (iv) the thiol groups (and not cross-linkages

formed between –SOH and $–NH_2$) are thought to be mainly responsible for the setting of wool fabrics in crabbing. A.T.P.

Torsional Properties of Single Wool Fibres I—Torque–Twist Relationship and Torsional Relaxation in Wet and Dry Fibres
T. W. Mitchell and M. Feughelman
Text. Research J., **30** (Sept 1960) 662–667

Direct measurements of torque–twist relationships for single wool fibres, both wet and dry, are consistent with a molecular model in which the matrix is a cross-linked polymer held together by a network of hydrogen bonds. C.O.C.

Kinetic Study of the Wool–Water System. II—Mechanisms of Two-stage Absorption
I. C. Watt *Text. Research J.*, **30** (Sept 1960) 644–651

The kinetics of the uptake of water vapour by wool depend on the initial concentration of water and the size of the concentration increment. For small concentration increments, absorption may occur in two stages, the second being much slower than the first. For any set of conditions, the previous treatment of the wool with respect to water vapour affects the kinetics and the equilibrium concentration of water in the fibres. The first-stage absorption to a quasi-equilibrium obeys Fick's laws of diffusion with a concentration-dependent diffusion coefficient. The apparent activation energy of diffusion varies with concentration. Second-stage absorption is accompanied by irreversible configurational changes within the fibres. In the first stage entry of water into the fibres causes swelling stress. In the second stage interchain bonds are broken, enabling the swelling stress to decay. At high regain the stress relaxation occurs quickly, following diffusion, and so no two-stage behaviour is seen. (For Pt. I see J.S.D.C., **76** (Nov 1960) 645.) C.O.C.

Sorption Kinetics of Water Vapour on Wool Fibres: Evaluation of Diffusion Coefficients and Analysis of Integral Sorption
P. Nordon, B. H. Mackay, J. G. Downes, and G. B. McMahon *Text. Research J.*, **30** (Oct 1960) 761–770

Simple diffusion theory does not explain the integral sorption kinetics for changes starting from dryness, the predicted uptake curves being steeper than those observed experimentally. This suggests that there may exist a slower rate-limiting process superimposed on the diffusion process. Speculations are made as to the nature of such a process. C.O.C.

Behaviour of Wool During Drying
D. Hildebrand *SVF Fachorgan*, **15** (Oct 1960) 617–622

After discussing general principles of drying, the author examines the effect of pH, moisture content, temperature and time of drying on the physical and chemical properties of wool. Damage during drying can occur if (*a*) wool is too alkaline, (*b*) its moisture content is too high and the drying time is too long, and (*c*) the fibre is over-dried. Conditions (*a*) and (*b*) result in the breakdown of cystine linkages and formation of lanthionine bonds. This is accompanied by the fall in the urea–bisulphite and alkali solubility of wool. Although the cystine content of the over-dried wool is not altered, its urea–bisulphite and trypsine solubility are decreased. Measurements of cystine and lanthionine contents, and of solubility in alkali and in urea–bisulphite are suitable for assessment of chemical modification. Their usefulness decreases if, before being dried, the wool was subjected to chemical treatments. The author concludes by comparing two commercial drying procedures at 125 and 135°C, respectively, both having no detrimental effect on the fibre. 27 References. L.A.T.

Softening of Raw Silk
W. Tsuji and M. Imai
J. Soc. Textile Cellulose Ind. Japan, **16** (Oct 1960) 879–884

Raw silk is softened considerably by various chemical treatments, e.g. with solutions of potassium rhodanate, $CaCl_2$, NH_4Cl, $NaHCO_3$, triethanolamine. The stiffness of the treated silk was measured by Euler's equation—

$$P_{max} = 4\pi^2 EI/l^2$$

(P_{max} = buckling force, measured by torsion balance, E = Young's modulus, I = moment of inertia, l = length of sample). The mechanism of softening is discussed and it is concluded that the raw silk is plasticised by the chemicals. A.T.P.

Chemical Attack of Textile Fibres. III—Synthetic Fibres
F. O. Howitt *J. Textile Inst.*, **52** (Jan 1961) P 14–P 27

Part III of a series, Part I (see *J. Textile Inst.*, **47** (1956) P 909) dealing with cellulosic fibres, and Part II (see J.S.D.C., **76** (1960) 380, 605) with protein fibres. Describes basic chemical reactions involved in preparation of polyamides polyesters, polyvinyl and polyvinylidene derivatives, polyethylene and polytetrafluoroethylene, and the action of chemicals on nylon, Terylene, Vinyon, Dynel, Saran, Orlon, Courlene, and Teflon. M.T.

Dependence of the Hygroscopic Equilibrium of some Man-made Fibres on Temperature
K. Windeck *Melliand Textilber.*, **41** (Nov 1960) 1407–1413

The influence of temperature on the hygroscopic equilibrium of some man-made fibres is investigated, the apparatus used enabling a reliable determination of the dry swelling of fibres to be made in a stationary atmosphere at any desired relative humidity and temperature. Full experimental details, and the results for the five fibres examined, are given. I.G.L.

Heating Orlon Acrylic Fibre to Render it Fireproof—Fiber AF
W. G. Vosburgh *Text. Research J.*, **30** (Nov 1960) 882–896

A black, fireproof fibre, Fiber AF, is prepared by heating Orlon in air at > 160°C. The fibre has an unusually high resistance to destruction by short-time heating, being over 100 times more resistant than ordinary fibres. Its abrasion resistance is poor. C.O.C.

Nylon II Fibres: Rilsan
P. A. Koch *Teintex*, **25** (15 Dec 1960) 888–897

Tabulated information on history, manufacture, constitution, properties, and bibliography. S.R.C.

Schreiner Calendering of Nylon Tricot
Bull. Centre Recherches Bonneterie, No. 12 (1960) 27–34

Schreiner calendering of nylon tricot locknit fabrics produces a marked increase in fabric opacity, improved handle, increased light reflectance and improved printability. Optimum results are obtained by calendering followed by heat setting before wet processing, e.g. scouring, dyeing. The effects obtained show a high degree of permanence to domestic washing treatments. The effects of variations in calendering conditions, i.e. roller pressures and temperatures, have been studied. G.E.K.

Glass Fibres
P. A. Koch *Textil-Rund.*, **15** (Oct 1960) 532–539

A tabulated summary of development, manufacture, properties, manufacturers' and patent literature (Fibre Table Series, FT7). S.R.C.

PATENTS

Mass Coloration of Polyamides
S *BP* 860,628 (Switzerland 24 Sept 1957)

Polyamides are mass coloured yellow-green to blue-green of outstanding fastness to light, gas-fumes, washing, perspiration, rubbing (crocking), cross-dyeing, sublimation, chlorination, and sodium chlorite bleaching by pigments obtained from the condensation of metal phthalocyanines containing 4–8 chlorine atoms with mercaptans in presence of an alkali-metal hydroxide and an alcohol, preferably benzyl alcohol. Thus, 30 parts of the condensate of 1 mol. octachloro-copper phthalocyanine and 8 mol. 4-methyl-1-mercaptobenzene (in presence of KOH and benzyl alcohol) is mixed with nylon 6.6 (5000) and the mass melted in N_2 at 285°C. The spun filaments are a yellowish green of outstanding fastness and may be orientated and/or crimped or shrinkproofed without detriment. A.T.P.

Surface Treatment of Polyesters
Eastman Kodak *USP* 2,921,828 (3 Dec 1956)

Films or fibres of highly polymeric high-melting linear polyesters derived from a bifunctional hexacarbocyclic *p*-dicarboxylic compound and a bifunctional dihydroxy compound are treated at elevated temperatures with one or more primary amino compounds, e.g. monoethanolamine, *N*-acetylethylenediamine, of general formula—

$$H_2N\left(\left[CHR^1{}_2\text{-}NR^2\right]_{y-1}\text{-}Z\right)_{m-1}\left(NR^2\right)_{n-1}$$

($x = 1$–10; $y = 1$–6; m and $n = 1$–2; $m + n = 3$; R^1 = H, Alk, OH, hydroxyalk; R^2 = H, Alk; R^3 = H, Alk, benzenearyl; Z = H, Alk, hydroxyalk, alkylcarbonyl, none of R^1, R^2, R^3, Z have > 10 C). The treated fibres have reduced tendency to static electrification, increased adherence to organic coatings, and increased receptivity for dyes, especially acid wool dyes. Thus, polyethylene terephthalate fibre is soaked in a solution of diethylenetriamine (5), and toluene sulphonic acid (1) in isopropanol (100) and pressed out to give 3% amine residue (dry basis). After heating for 10 min at 150°C (in mineral oil), the modified fibre has excellent uptake of acid wool dyes. A.T.P.

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Removal of Oil from Textiles

J. C. Stewart and C. S. Whewell

Text. Research J., **30** (Dec 1960) 903–912

Trials made using paraffin and olive oil on a wide range of fibres and Lissapol N (ICI) as detergent showed difficulty of oil removal to be associated with matching of the polarities of oil and fibre which, since oils are at least moderately non-polar, means that in general the more hydrophobic the fibre, the more it is difficult to remove oil. A general correlation exists between detergency and contact-angle measurements, which indicates the adequacy of the "rolling up" mechanism as a description of the mechanism of detergency for these two oils on loose fibres. 30 References. C.O.C.

Removal of Oil from Wool and its Relationship to Surface Structure

J. C. Stewart and C. S. Whewell

Text. Research J., **30** (Dec 1960) 912–918

It is difficult to remove oil from undamaged wool because the fibre has an epicuticle which is mainly paraffinic in nature. The polarity of the fibre surface, ease of oil removal, and the degree of completeness of the epicuticle are directly related. Treatments which make wool readily washable do so by removing the hydrophobic epicuticle and so exposing the underlying hydrophilic keratin. 29 References. C.O.C.

Discoloration of Wool Grease in the Recovery Process

C. A. Anderson and E. V. Truter

J.S.D.C., **77** (Feb 1961) 63–64

Newer Developments in Sodium Chlorite Bleaching

O. Deschler

Canadian Textile J., **77**, No. 25 (25 Nov 1960) 62–68

The development of bleaching in Europe, particularly continuous open-width bleaching using sodium chlorite, is discussed. More than half the sodium chlorite supplied to industry is used for bleaching cotton. The advantages of open-width bleaching using sodium chlorite, particularly even treatment, saving in time, and absence of degradation, are emphasised. The overcoming of corrosion of equipment is stressed. Three different types of continuous open-width bleaching installations in use in Europe are described. W.P.M.

Sodium Chlorite Bleaching

H. Hefti

I— Decomposition Studies

Text. Research J., **30** (Nov 1960) 861–867

In absence of oxidisable matter the decomposition of sodium chlorite can be fully explained by the equations—

$$5\ ClO_2^- + 2\ H^+ \rightarrow 4\ ClO_2 + Cl^- + 2\ OH^- \quad (I)$$

$$3\ ClO_2^- \rightarrow 2\ ClO_3^- + Cl^- \quad (II)$$

$$ClO_2^- \rightarrow Cl^- + 2\ O \quad (III)$$

I was always the main reaction in the tests made, II consumed small amounts of chlorite in three cases and was of some importance in presence of Al sulphate, III occurred in all tests but never consumed > 10% of the chlorite decomposed. I indicates that ClO_2 formation is controlled by the hydrogen ion activity of the solution. No general rule could be established to connect pH with the hydrogen-ion activity of the different activators used. The relative amounts of chlorite used for forming ClO_2 decrease with decreasing pH. Formation of chlorate occurs in presence of diethyl *d*-tartrate, NH_4Cl, triethanolamine hydrochloride, and Al sulphate; with the exception of Al sulphate, measurable amounts of chlorate were formed only at pH < 5. Chlorate formation generally increases with decreasing pH. All the activators tested gave small amounts of oxygen, the amounts of chlorite used for this being 6–9% of the total decomposed and only slightly dependent on pH. Decrease in pH increases the rate of chlorite decomposition, thus decreasing the time required for bleaching.

II— Bleaching Studies

Ibid., 867–872

Neutral chlorite solutions give acceptable bleaching at ≤ 100°C. The possibility of seed husks and waxes acting as activators was excluded, but apparently cellulose may be an activator at > 100°C. In any case, bleaching is dependent on temperature and at 130°C no activator is needed to achieve good bleaching, but at < 100°C presence of an activator does have good results. Practically no tendering occurs in chlorite bleaching. Corrosion of plant exposed to the liquor is almost completely eliminated by adding $NaNO_2$ to the bleach liquor. Corrosion by gases can be reduced by using activators which liberate no ClO_2 or still better with NH_4 salts, which react with alkaline chlorite solution as follows—

$$NH_4^+ + OH^- \rightarrow NH_3 \uparrow + H_2O$$

These salts appear to improve bleaching, at least at 100°C. C.O.C.

Effect of Hypochlorite Bleaching Variables on Properties of Prehydrolysed Sulphate Hardwood Pulp

J. W. Smith and W. L. Thornburg

Tappi, **43** (June 1960) 596–599

Initial pH is the only variable in the hypochlorite bleaching stage affecting every pulp property at 99% confidence level. Temp. and % available chlorine have very similar effects on the various properties, while the effect of time is appreciably smaller and the initial viscosity in the hypochlorite bleaching stage has the least influence. Solubilities (determined by Swedish standard CCA 8:55) reach a max. when 19·5% active alkali is used in the cook and do not change appreciably during bleaching. R.A.

The Stabiliser in Hydrogen Peroxide Bleaching

D. M. Cates and W. H. Cranor

Text. Research J., **30** (Nov 1960) 848–855

Reflectance and chemical attack in the fibre are functions of the amount of H_2O_2 decomposed and both are relatively unaffected by the rate of decomposition either in presence or in absence of a stabiliser. For a given amount of decomposition, stabilised peroxide yields better reflectance than the unstabilised, but also causes more damage. However, there is no difference in reflectance achieved per unit reduction of viscosity. When scoured fabric is treated to remove traces of metals, the rate of decomposition of stabilised peroxide is appreciably reduced. Such treated fabric is more effectively bleached than untreated scoured fabric for the same amount of peroxide decomposed, but it also suffers more damage. In terms of reflectance achieved per unit reduction of viscosity, better results are obtained by removing all traces of metal from the fibres. C.O.C.

Mercerising and Wetting Agents— II

G. Dollinger, M. Mohaupt, and M. Rösch

Melliand Textilber., **41** (Nov 1960) 1398–1403

(For Pt. I see J.S.D.C., **76** (May 1960) 322.) Factors affecting the lustre obtained in the mercerisation process, and the influence of synthetic wetting agents are discussed. From the experiments described, it is concluded that the use of a wetting agent is essential if satisfactory results are to be obtained in crude mercerisation, this being of course dependent on the choice of the most suitable mercerisation period, temperature range, and optimum concentration of the wetting agent. The choice of agent for the mercerisation of hank and piece is also considered, a slow wetting and shrinking product being preferable in the case of hanks, and an auxiliary product of rapid wetting and shrinking properties being necessary for piece mercerisation. Attention is also drawn briefly to the advantages and disadvantages of the application of wetting agents based on cresol. I.G.L.

Brightening of Groundwood (XI p. 188)

VIII—DYEING

Experimental Investigation of Mechanical Factors in Dyeing

J. L. Häusermann *Textil-Rund.*, **15** (Aug 1960) 413–422

A number of factors, including direction of flow, pressure in padding, and vibration, were investigated on a small scale. S.R.C.

Coverage of Immature Cotton on Dyeing with Azoic Dyes

H. P. Maier *Melliand Textilber.*, **41** (Nov 1960) 1384–1389

The origin and effect of neps and knops in cotton fabrics and their coverage on dyeing with azoic dyes is discussed. Their visibility after dyeing has been shown to be dependent on the concentration of the azoic coupling component in the dyebath, being greatest at ca. 5 g/l. Pretreatment of the cotton fabric also affects coverage on subsequent dyeing with azoic dyes, e.g. the effect of desizing agent, bleaching conditions, concentration of caustic soda in the pretreatment of poor-quality fabrics before dyeing. Experiments on protein cleavage are discussed briefly. I.G.L.

Application of Reactive Dyes to Knitwear—Dyeing Cellulosic and Polyamide Fibres

J.-J. Prévot

Bull. Centre Recherches Bonneterie, No. 12 (1960) 21–25

The chemistry of the reaction between a reactive dye and a cellulosic fibre is briefly discussed. Application of the mono- and di-chlorotriazine types, e.g. Procion and Procion H brands, by the exhaustion technique, including methods of control for obtaining adequate penetration of tightly twisted yarns, is described. The importance of using electrolytes, i.e. sodium chloride and sulphate, free from impurities such as salts of calcium, magnesium, and aluminium, which result in precipitation of the dye, is emphasised. The application of Procinyl dyes to polyamide films, producing dyeings of high wet fastness and covering power equal to that of disperse dyes, is also described. G.E.K.

Suspension Method for Dyeing Cotton and Viscose Fabrics

K. M. Shostko and T. T. Rozinskaya

Tekstil. prom., **20** (Jan 1960) 50–52; (Feb 1960) 46–50

This is a modified method of application of direct dyes to cotton and viscose rayon fabrics, and is claimed to shorten the process by 25% and considerably reduce the consumption of steam and electricity. The dyeing process consists of two stages. (1) The preparation of a very fine suspension of an insoluble Ba salt of a direct dye by the addition of $BaCl_2$ to the dye soln. at 40–45°C. $2R{\cdot}SO_3Na + BaCl_2 \rightarrow (R{\cdot}SO_3)_2{\cdot}Ba + 2NaCl$. During heating to 90°C the fine Ba salt particles are absorbed by the fibre. (2) Dyeing proper involves hydrolysis of the salt with the liberation of the free dye, which diffuses into the fibre. This stage is completed during 10–15 min at the boil. To ensure level results it is important to raise the temp. gradually. Dyeings so produced are of a very high quality as regards tone and evenness of shade. Most direct dyes are suitable for application by the suspension method. A few give better results with $MgCl_2$. Bleaching with hydrogen peroxide and a silicate in presence of a wetting agent can be incorporated in the dyebath. T.Z.W.

Dyeing Viscose Rayon–Wool Mixtures by an Acid One-bath Method

W. Festerling *Chemiefasern*, No. 1 (Jan 1961) 51–55

The dyeing of viscose rayon–wool unions by a one-bath process in acid medium is reviewed. It is considered preferable to the neutral one-bath method, the disadvantages of which are briefly discussed. A pH range of 4–5 is recommended, and dyes suitable for the production of solid shades and two-tone effects are given, with details of further dyebath additions (resists, levelling agents). Examples of dyeing procedure are given for solid shades and two-tone effects on hank and cheese, and for the production of a two-tone effect on upholstery material. The method is considered to be of the greatest importance for the union dyeing of wool and mohair plush. I.G.L.

Continuous Dyeing of Wool by Means of an Aqueous Two-phase System; Coacervation as a New Concept in Textile Processing

R. Casty *Canadian Textile J.*, **77**, No. 25 (25 Nov 1960) 47–61

After a brief discussion of processes previously advocated for dyeing wool continuously, the principle of coacervation and its application to continuous dyeing, particularly of wool, are described in detail. Reaction products of fatty acids with an excess of alkylol amines (e.g. Cibaphasol C) readily promote coacervation. Dyes and pigments, unless extremely hydrophilic, favour the coacervate (which forms a large proportion of the total volume of the system) almost exclusively. Separation of the emulsion into coacervate and dilute equilibrium liquid occurs quickly, and a thickener (sodium alginate) is necessary to give a homogeneous liquid suitable for padding purposes. Suitable pad-liquors contain 40 g/l. Cibaphasol C, 50–300 g/l. sodium alginate (2·5% solution) and x g/l. dye. Wool may be dyed by the continuous pad–steam or by pad–roll processes. The coacervate forms a continuous layer on the wool and has no affinity for the fibre, and the dye does not migrate into the fibre until it is steamed. Consequently there is no "sandwich" effect and no skitteriness. Acid, prometallised, and chrome dyes may be applied, and dyes of different groups are miscible with each other. The process causes no greater reduction in cystine content and tensile strength of the wool than do conventional dyeing methods. Wool–polyamide, wool–cellulosic and wool–polyester unions have been dyed by this method. Suitable machinery is suggested. W.P.M.

(This article has also appeared in the following journals — *Amer. Dyestuff Rep.*, **49** (12 Dec 1960) 952–964; *Dyer*, **125** (27 Jan 1961) 100–112 (abbrev.); *Melliand Textilber.*, **41** (Nov 1960) 1365–1376; *SVF Fachorgan*, **15** (Dec 1960) 822–835; *Teintex*, **26** (Jan 1961) 7–25; *Tinctoria*, **57** (Nov 1960) 443–453— Ed.)

Kinetics of Wool Dyeing—Some Effects of Alcohols on Dyeing Rates

J. A. Medley and M. W. Andrews

Text. Research J., **30** (Nov 1960) 855–860

Wool fibres behave as homogeneous, infinitely long cylinders with a surface barrier when dyed with the free acid of Orange II (C.I. Acid Orange 7). The accelerating action of butanol on the rate of dyeing is connected with a modification of the dyeing mechanism which has its main effect inside the fibre. Butanol increases the diffusion rates reversibly by this modification, and also irreversibly, probably by removing material from inside the fibre. Ethanol also produces this irreversible effect. The over-all effect is to make the surface barrier, or surface resistance to dyeing, more significant than when dyeing from water alone. C.O.C.

Mode of Reaction of Procinyl Dyes

E. Elöd and V. Einsele

Melliand Textilber., **41** (Nov 1960) 1377–1384

The Procinyl dyes are compared with those classes of dye previously available for dyeing polyamide fibres, and evidence for bond formation between the reactive dye and fibre is discussed. Further evidence for this linkage is found in the results of diffusion experiments, and in the chromatography of dyed polyamide fibres. The dependence of dye uptake and the amount of dye fixed to the fibre on the amino-group content of the fibre is shown. Further, blocking the amino group by acetylation or deamination was found to reduce dye uptake; conversely, increasing the amino-group content resulted in increased dye uptake. The possibility of there being some reaction between dye and the acid amide groups is discussed, support for this being found in the results of dyeing experiments with model substances (urea resin and polyurethanes). The ratio of dye taken up to that fixed to the fibre was found to vary, decreasing in the order wool, silk, Perlon, urea resin, and polyurethane. Uptake of dye by Perlon is independent of pH, but uptake by wool and silk increases with increasing pH. At pH 10, both wool and silk take up and combine with more dye than does Perlon. In dyeing of mixtures of Perlon and protein fibres in alkaline medium, the latter take up more dye. Dyeings of all fibres showed good penetration. I.G.L.

Constitution and Effectiveness of Organic Substances as Carriers in the Dyeing of Polyester Fibres with Disperse Dyes—III

P. Senner *Textil Praxis*, **15** (Nov 1960) 1168–1171

For Part I see *ibid.*, **15** (Sept 1960) 940 (J.S.D.C., **77** (Jan 1961) 42); For Part II see *ibid.*, **15** (Oct 1960) 1051 (J.S.D.C., **77** (Feb 1961) 83). The author examines critically various theories of the mechanism of carrier action— (1) Disperse dyes are more soluble in the carrier phase, and

form a higher concentration gradient at the fibre–liquor interface. Consequently the diffusion of dye molecules into the fibre is facilitated. (2) The formation of a dye–carrier complex cannot be generally accepted because several substances with good "carrier" properties are perfectly symmetrical and their dipole moment is zero. In addition, there is no reason why a large complex should diffuse more readily than a smaller dye molecule. (3) The possibility of formation of a ternary union of fibre–dye–carrier analagous to a eutectic mixture is not very likely. (4) It is possible that the carrier acts as an internal "lubricant" for the fibre, and consequently dye molecules require less activation energy for the diffusion process. (5) The view favoured by the author is that the function of the carrier is to form channels and cavities, thus facilitating access of dye molecules into the interior of the fibrous matter. The effectiveness of the carrier is governed by its molecular constitution, and steric factors play an important part. L.A.T.

Dyeing Vinylon with Solacet Dyes
A. Katayama, N. Kuroki, and K. Konishi

I— Relation between Affinity and Salt Concentration of the Dyebath
J. Soc. Textile Cellulose Ind. Japan, **16** (Dec 1960) 1041–1046

The absorption by Vinylon of Solacet dyes (Fast Orange 2GS, Fast Blue 2BS, Fast Scarlet B, Fast Yellow GS, and Fast Crimson BS) has been studied over a wide range of dye and salt concentrations at 90°C. The addition of electrolyte promotes dye absorption, except with Fast Scarlet B, when it has no effect. The standard affinity of the dyes for Vinylon is −3 to −6 kcal/mole. This class of dye, despite small molecular size, shows considerable affinity. The affinity is reported to be fairly constant under any dyeing conditions with Fast Orange 2GS and Fast Crimson BS, whereas affinity tends to increase with increasing salt concentration in the case of Fast Yellow GS and Fast Blue 2BS. However, at salt concentrations of up to 10 mmole/l., increase of salt concentration was found to result in a decrease in affinity in all cases. This abnormal dyeing behaviour is discussed.

II— The Heat and Entropy of Dyeing
Ibid., 1046–1049

The absorption of Solacet dyes by Vinylon has been studied at various temperatures. The heat of dyeing is −5 to −12 kcal/mole, and the entropy of dyeing −1 to −15 cal/mole/degree. On comparison with the direct dyes, the heat of dyeing is seen to be slightly less, and the entropy of dyeing considerably smaller. The fact that the affinity of the Solacet dyes is similar to that of the direct dyes is attributed to the small value of the entropy term. I.G.L.

Dyeing Anodised Aluminium
C. Th. Speiser *Textil-Rund.*, **15** (Oct. 1960) 540–548

A review of mechanical and technical preparation, oxidation, dyeing with inorganic and organic dyes and sealing. S.R.C.

PATENTS

Dyeing Polyesters Based on Terephthalic Acid
FH *BP* 855,546 (Germany 10 Mar 1956)

Deep dyeings of excellent fastness to rubbing are obtained if the polyesters are first treated with addition products of ethers or tertiary amines with SO_3 and/or halides of sulphur oxy-acids in presence of inert solvents. Thus stretched staple fibres of polyethylene glycol terephthalate, after refluxing with chlorosulphonic acid and dioxan in ethylene chloride, readily yield deep dyeings with basic and disperse dyes. The dyeings are clear, of excellent fastness to rubbing and very good fastness to light and to washing. The fastness to sublimation and to hot ironing is improved by aftertreating with a solution of hydrazine or an *N*-formyl compound having a reactive H atom on the N atom carrying the formyl group. C.O.C.

Dyeing Anodised Aluminium
Colonial Alloys Co. *USP* 2,927,872 (19 Dec 1957)

Dyeings of good fastness to light are obtained by dyeing for a few minutes in a bath containing an anthraquinone acid or mordant dye and a hydrolysable salt of a weak metallic base, e.g. Ni acetate or ferric ammonium oxalate. Dyeing is followed by sealing with a hydrolysable salt of a weak metallic base, if desired the same salt that is used in the dyebath. Alternatively, sealing with a mixture of Ni acetate and a soluble fluoride renders unnecessary presence in the dyebath of a hydrolysable metal salt. C.O.C.

Reducing Agents for Use in Vat and Sulphur Dyeing (III p. 171)
The Colour Behaviour of 6-(2-Hydroxy-3-naphthamido)-1-naphthol-3-sulphonic Acid (IV p. 173)
Influence of Soaping on Thioindigoid Dyes (IV p. 175)
Mass Coloration of Polyamides (VI p. 182)
Effect of Preparation and Type of Dye on Leather Dyeing (XII p. 188)

IX— PRINTING

Experimental Preparation of Thickeners from Xylose
Chin-Lin Lu *Jang Hua*, (8) (1956) 35–36
Referat. Zhur., Khim., (1958) Abstr. No. 16570
Chem. Abs., **54** (25 Nov 1960) 25838

Use of thickeners made from xylose promotes production of better colour values when printing with vat dyes. The nature of the starting materials and the method of xylose preparation have marked influence on the thickener quality. Xylose is obtained by hydrolytic cleavage of vegetable materials containing large amounts of xylan. With rice straw NaOH is used, and with cane $ClCH_2COOH$. C.O.C.

PATENTS

Printing with Water-in-oil Emulsions of Vat Dyes
DuP *BP* 856,325 (U.S.A. 12 Sept 1956)

A water-in-oil emulsion is formed by adding water to a water-immiscible hydrocarbon solvent containing a caustic alkali-permeable resin dissolved and/or colloidally dispersed in it. The resin forms 1·0–4·5% by wt. of the emulsion. To this is added ⩾ 3% by wt. of an aqueous vat dye paste containing a dispersing agent. The resulting emulsion is applied on conventional equipment using shallow engraved intaglio rolls which may be run at higher speeds when this thickened paste is used. The prints are dried and then passed through an alkaline hydrosulphite bath into an air-free steamer to reduce and fix the dye, which is then reoxidised in the usual manner. C.O.C.

Thickening Agents for Printing Pastes Containing Reactive Dyes
W. A. Scholten's Chemische Fabrieken *BP* 856,284 (12 Aug 1958)

Water-soluble ethers and/or esters of starch containing 0·5–1·5 ether and/or ester groups per anhydroglucose unit, but free from free hydroxyl or amino groups, are good thickening agents for use with reactive dyes. They are readily washed out of the fabric, leaving it soft and supple. They give a higher colour yield than alginates. C.O.C.

Colour Printing
J. F. Crosfield *BP* 855,895 (11 Mar 1957)

Describes a method of producing a colour-corrected colour-component printer for accurate reproduction of coloured originals. C.O.C.

Transfer Sheets
Columbia Rubber & Carbon Mfg. Co. *BP* 855,912 (14 Jan 1957)

A flexible base is coated with coloured pressure-transferable material. A topcoating is then applied which is also pressure-transferable and contains sufficient pigment to give it a colour contrasting strongly with that of the main transferable material. Thus after use the areas which have been transferred are clearly visible. C.O.C.

Typewriter Ribbons
Caribonum *BP* 856,188 (2 Aug 1957)

Typewriter ribbon impregnated with an *N*-substituted mono- or di-leucauramine dissolved or suspended in an oil is colourless but yields coloured characters when used with paper containing an acid-fixing paper, e.g. the leucauramines and papers described in *BP* 852,131 (J.S.D.C., **77** (Feb 1961) 86). C.O.C.

Yellow Colour Couplers (IV p. 175)
Light-sensitive Diazonium Salts (IV p. 176)
Antistatic Photographic Films (XI p. 188)

X— SIZING AND FINISHING

Control of Exhaust Air Humidity During Drying of Textile Fabrics

R. Burgholz *Textil Praxis*, **15** (Nov 1960) 1151–1157

The author gives an account of his experience, under works conditions, of factors influencing the economics of drying of textile fabrics and the performance of commercial dryers. Generally speaking, the temperature and humidity of the fabric, the drying room, and the exhaust air duct determine the efficiency of drying. The drying process is more economical at higher temperature and humidity, the velocity of the drying medium being so adjusted that the moisture content in the exhaust air remains uniform.

L.A.T.

Self-smoothing Fabrics— The Triazines

J. T. Marsh *Text. Manuf.*, **87** (Jan 1961) 25–27

The derivation of, and relationship between, the various triazine compounds used in this field and other reagents is briefly reviewed. The advantages and disadvantages of the methylolmelamines is discussed, with particular reference to chlorine retention. At the present time considerable interest is being shown in the triazines as crease-proofing agents, but more use is being made of their valuable properties as part of a formulation than as simple chemical reagents. S.B.D.

Relationship between Fibre Properties and Fabric Crease-recovery

W. W. Daniels *Text. Research J.*, **30** (Sept 1960) 656–661

For many fibres a one-to-one correspondence was found between fabric crease-angle recovery and a modified tensile recovery parameter, i.e. average value of tensile recovery (over the range of strains imposed during creasing) measured against a restraining force related to the coefficient of interfibre friction. From this index of recovery it was predicted that fabric bending recovery would tend toward zero with decreasing strain (increasing values of crease curvature). This proved to be true with nylon and Dacron fabrics. Tensile recoveries under load, effective frictional restraints, and qualitative fabric-recovery features indicate that the predominant fibre properties controlling fabric recovery can be determined without recourse to a complete mathematical treatment. Interfibre friction must be considered in order to obtain even a qualitative description of the mechanism of fabric recovery from deformations involving small strains.

C.O.C.

Studies on the Resin Distribution during Resin-finishing of Fabrics

VI— Effect of Added Thermoplastic Resin

S. Yokoyama and Y. Onti

J. Soc. Textile Cellulose Ind. Japan, **16** (Oct 1960) 866–870

Added thermoplastic resin (I) decreases the migration of the precondensates of thermosetting resins at the pre-drying stage in the resin finishing of cellulosic fibres. It is concluded that this controlling effect of I on the resin distribution is an important factor in the increased tear and abrasion strength of the finished fabric (in addition to the previously recognised softening action).

VII— Effect of Infrared Heating

S. Yokoyama *Ibid.*, 870–872

Predrying was carried out by infrared heating in an attempt to avoid the resin migration occurring in hot-air predrying (heat conduction into the fabric). With hot-air heating, migration was extensive as the pick-up (%) increased, but with infrared heating, migration was not noticeable at a pick-up of 70–90%. A.T.P.

Studies on the Resin Distribution during Resin-finishing of Fabrics using Radioactive Isotopes

S. Yokoyama and Y. Onti

I— The Fundamental Conditions of Macroauto-radiography

J. Soc. Textile Cellulose Ind. Japan, **16** (Dec 1960) 1050–1053

As the first step in the radio-isotopic investigation of resin distribution during resin finishing of fabrics, the optimum conditions for obtaining macroautoradiographs of the finished fabrics were determined. Details are given.

II— Effects of Predrying Conditions on Resin Distribution

Ibid., 1053–1055

By the application of autoradiography, it is concluded that (*a*) the lower the predrying temperature, the weaker is the migration of bath solids, (*b*) the migration of bath solids is considerably reduced by infrared predrying, and (*c*) if, during the predrying, the fabric comes into contact with metals, the bath solids migrate, resulting in fluctuation in resin content. This fluctuation is not affected by soaping and results in non-uniform fabric properties. I.G.L.

Improved Textile Properties from New Chemical Structures

A. M. Sookne

Amer. Dyestuff Rep., **49** (28 Nov 1960) P 890–P 896

In recent years fibre producers have provided an array of fibres with new chemical structures and improved textile properties resulting from increased crystallinity, as in the isotactic polyolefins, improved orientation, or the use of graft polymers. The textile chemist in his finishing operations also changes the structure of the fibres, and the relationship between chemical structure and the useful textile properties of such chemically modified fibres is discussed. New evidence is presented that, for "wash-and-wear" treatments with vinyl cyclohexene dioxide, the introduction of intermolecular cross-links is largely responsible for the improved properties of cotton fabric.

W.G.C.

Crease-resistant Treatments for Cotton

B. C. M. Dorset

Text. Manuf., **86** (Dec 1960) 495–499, 503

Some recent developments in crease-resistant treatments for cotton fabrics are reviewed and supported by original data. Drip-dry finishes for cotton and wool are aimed at reducing the hydrophilic nature of the fibre and employ typical crease-resistant resin finishes developed for improving the crease resistance of dry fabrics. In drip-dry finishes, however, both wet and dry crease resistance are desirable. Wet crease resistance can be improved by treatment of cotton fabrics with formaldehyde (7·4% concn.) in 5-N hydrochloric acid for 30 min at 28°C, with a loss in tear and tensile strengths of the same order as found in conventional crease-resist treatments. Virtually no improvement in dry crease resistance occurs, however, so that a subsequent minimal treatment with an amino-formaldehyde resin is suggested. Dry crease resistance can also be improved by treatment with an organic diepoxide, e.g. vinyl cyclohexene diepoxide, and gives fabrics free from chlorine retention and of good resistance to acid treatment and repeated washing. Recently the condensation products of formaldehyde with the dihydrazides of aliphatic dicarboxylic acids have been shown to be effective in the crease-resist treatment of cotton, giving a relatively small fall in tensile strength but average reduction in tear strength. Chlorine retention of these products is much less marked than with the conventional amino-formaldehyde resins currently employed. Data are also given on the slow release of formaldehyde during storage of fabrics treated with a range of amino-formaldehyde resins owing to inadequate washing out of catalyst residues after curing. E.C.

Polymerisation and Condensation Reaction of *N*-Methylolacrylamide in Cotton Cloth

H. Kamogawa, R. Murase, and T. Sekiya

Text. Research J., **30** (Oct 1960) 774–781

Crease resistance and, under certain conditions, abrasion resistance were excellent in cloths treated with *N*-methylol-acrylamide, which was then polymerised. Temperature and duration of predrying and the medium (water or organic solvent) of the treating bath had some slight effect, possibly because of formation of rather weakly cross-linked polymer within the fibres and cross-linking between the polymer and the cellulose. Addition of comonomers, e.g. acrylamide or hexamethylene diamine hydrochloride, or softeners to the bath was effective in reducing loss in strength. Aftertreatment with hexamethylene diamine was also effective, and better results were obtained if this was followed by treatment with formaldehyde. C.O.C.

Cross-linking of Cotton Cellulose. I—Chemical and Physical Aspects

F. S. Perkerson, W. A. Reeves, and V. W. Tripp

Text. Research J., **30** (Dec 1960) 944–954

The types of cross-linking agents now used on cotton and the resulting finishes are discussed, and it is concluded that either the ideal agent has yet to be found or the best method of application has yet to be discovered. Cross links through primary covalent bonds are largely responsible for the physical changes in cross-linked cotton, but secondary valence cross-links may also produce significant changes, some of which are durable. Polymerisation with simultaneous cross-linking improves the over-all minimum-care properties, although polymer formation alone does not improve crease recovery. To produce crease-recovery angles in the practical range, larger amounts are needed of cross-linking agents capable of forming thermosetting resins than of agents which do not form such resins. The extent of permanent set is related to the extent of cross-linking. Regain and fibre density are more dependent upon the degree of fibre swelling at the time of cross-linking than upon the amount of cross-linking. C.O.C.

Cross-linking of Partly Cyanoethylated Cotton. III—Acid Scouring of Amidoximate and Subsequent Formaldehyde-curing

M. Negishi, H. Itō, and K. Takahashi

Text. Research J., **30** (Dec 1960) 999–1003

Scouring with 0·1–0·5% HCl and then curing with neutral formaldehyde in absence of a catalyst and with or without tension much increased the crease resistance of amidoximates of partly cyanoethylated cotton fabrics without much loss in tensile strength, elongation, and tear strength. The handle was as soft as that of unmodified cotton. Apparently the amidoxime hydrochloride is formed during the acid scour and then during curing the HCl is split off and acts as a catalyst in ending intermolecular cross-linking. C.O.C.

Imparting Crease Resistance to Cotton with 1,1′-Carbonylbisaziridine

L. H. Chance, R. M. Perkins, and W. A. Reeves

Text. Research J., **30** (Dec 1960) 918–925

High crease resistance was obtained on cotton cloth by padding with aqueous 1,1′-carbonylbisaziridine, $(NCH_2–CH_2)_2CO$, in presence or absence of Zn fluoroborate as catalyst, drying and baking. This formed an insoluble resin in the fibre and probably also effected cross-linking of the cellulose by formation of ether linkages. The treated fabric had a good handle and appearance. The amount of resin used was the same as in other resin finishes. Tear and breaking strengths of cloth treated in absence of a catalyst were comparable with those obtained by most other methods. The strength and colour of treated cloths were impaired by a chlorine bleach and scorch when no catalyst was used in the resin treatment. When Zn fluoroborate was used as catalyst and the solution applied immediately after addition of the catalyst, no loss in strength resulted from a chlorine bleach and scorch, but discoloration still occurred in the scorched areas. The finish was very resistant to home laundering and boiling alkaline solutions. C.O.C.

Infrared Examination of Cellulose Hydrate Films Treated with Finishing Agents. II—Application of Modern Types of Reactant to Deuterated Cellulose Films

W. Bandel *Melliand Textilber.*, **41** (Nov 1960) 1391–1397

Infrared-spectrographic investigations have been carried out in order to explain the reaction of finishing agents with cellulose hydrate. Films were treated with various resin finishes by normal methods. Evidence for the formation of a bond between finishing agent and cellulose is discussed. In the work described, six commercial crease- and shrink-resist agents were examined. I.G.L.

Partly Cross-linked Viscose Rayon Nitrate

T. Fujimura and S. Okamoto

Text. Research J., **30** (Oct 1960) 796–797

Nitration followed by acetylation improves the crease recovery and water resistance of viscose rayon. C.O.C.

Studies on the Compressive Shrinkage of Spun-rayon Fabrics

T. Owashi and K. Senda

J. Soc. Textile Cellulose Ind. Japan, **16** (Oct 1960) 859–865

The compressive shrinkage of several cotton and rayon fabrics (all having same yarn count, twist, ends, and picks/cm) was investigated, and comparison was made between residual shrinkages and physical properties of fabrics after treatment. Residual shrinkage after a compressive shrinking process was in order, cotton (none) < spun-rayon (crumpled) < cotton-like rayon < regular rayon and super rayon (Avisco XL) (much). Fabrics with large residual shrinkages generally had poor dimensional stability (especially in length difference between conditioned and wet fabric) and not so much relaxation and progressive shrinkage. Broad cloths, which have large residual shrinkages, are made from fibres of high water imbibition and low wet modulus. Spun-rayon fabrics elongate more than cotton fabrics under identical loads in the standard state, and it is possible that unknown factors exist, as a result of which residual shrinkages of spun-rayon fabrics are not removed, even after application of a compressive process, e.g. Sanforizing. A.T.P.

Setting Wool with Ethanolamine

N. H. Koenig, W. L. Wasley, and C. E. Pardo

Text. Research J., **30** (Nov 1960) 901–902

Highly effective pleating of wool cloths is obtained by steam pressing them after treatment with 0·25–2·0% aqueous ethanolamine. C.O.C.

Reducing the Felting Power of Wool with Polyglycine from the Polymerisation of Anhydrocarboxyglycine

J. H. Bradbury and D. C. Shaw

Text. Research J., **30** (Dec 1960) 976–982

Anhydrocarboxyglycine cannot be used in aqueous solution because it is unstable in presence of water. Wool treated with 2% of its weight of anhydrocarboxyglycine for 1 h at 50°C or 7 h at room temperature in water-washed white spirit or trichloroethylene has its felting power satisfactorily reduced. The polymerisation, initiated by water in the wool, produces polyglycine of degree of polymerisation about 30. Descaling of treated fibres showed all the polyglycine to be on the outside 10% by weight of fibre; a surface layer can be seen by Sellotape or electron microscopy. It is this surface layer which is responsible for the large increase in the frictional coefficients μ_1 and μ_2 and decrease in $\mu_1 - \mu_2$, which in turn affects feltability. When ⩽ 0·39% polyglycine is used, some reduction in felting power is produced by spots of polyglycine randomly distributed over the fibre without appreciable masking of the scale edges. C.O.C.

PATENTS

Bulking Thermoplastic Synthetic-polymer Yarns

Ernest Scragg & Sons *BP* 850,693 (11 July 1957)
BP 857,916 (5 Apr 1958)
BP 857,967 (16 July 1958)
BP 857,974 (25 Nov 1958)
BP 858,438 (16 Apr 1958)

A twist-crimped yarn is relaxed, heated and cooled. Suitable machinery is described. C.O.C.

Elasticising Cellulose Ester Yarns

Deering Milliken Research Corpn. *USP* 2,925,641 (9 Feb 1956)

Greater permanency of the bulking effect is produced if, before treatment by the process of *BP* 558,297 (J.S.D.C., **60** (1944) 130), the yarn is treated with a reagent which increases its elongation at break without appreciably diminishing its strength, e.g. a non-oxidising mineral acid or a salt thereof, particularly $ZnCl_2$. C.O.C.

Zirconium Acetate–Zinc Acetate Catalyst in Silicone Proofing

Dow Corning Corpn. *USP* 2,927,870 (28 Aug 1956)

A mixture of Zr and Zn acetates having 0·1–4·5 Zn atoms per Zr atom used as a catalyst in silicone emulsions yields relatively stable emulsions which after baking, e.g. at 200°F for 10 min, need no ageing to develop full water-repellency. C.O.C.

Protecting Textiles, Furs, and Skins against Textile Pests, Moulds, and Bacteria
FBy *BP* 856,452 (Germany 27 June 1957)
The materials are impregnated with a halogenoalkyl-, halogenoaralkyl-, and/or halogeno-sulphonamide of an aminodiphenylamine or *N*-imino-substituted aminodiphenylamine which is halogenated in either or both aromatic nuclei, e.g. pentachloro-chloromethanesulphonyl-2-aminodiphenylamine, which can be used either in an organic solvent or as its Na or other salt in aqueous solution. C.O.C.

Ribbons
Jersey Fabrics *BP* 856,440 (19 Dec 1957)
A wide fabric is made which can be subdivided into ribbons by removing yarn located in the positions where divisions are to occur, such yarn being soluble in solvents which do not affect the bulk of the material. This enables the fabric to be dyed and finished as a wide fabric and then be batched on a perforated cylinder where it is treated with the solvent so that the ribbons may be separated. C.O.C.

Patterning Knitted Fabric
English Rose *BP* 849,676 (24 June 1958)
Ladies' nylon hose are permanently patterned for ornamentation or trade-mark purposes by penetrating the fabric with a pattern arrangement of heated needles, permanent enlargement of the stitches pierced by the needles being produced. G.E.K.

Metallised Fabrics
British Cotton Industry Research Assocn.
BP 856,341 (30 Nov 1956)
A thin sheet of metal is applied to the surface of a fabric and pressed into place by a resiliently deformable material. The pressure is such that the metal is made to conform to the surface contours of the fabric, to rupture at a sufficient number of the interstices of the fabric to give the required porosity, and to cause the ruptured parts of the sheet to extend into and conform to the wall contours of the interstices beneath them. C.O.C.

Ornamental Effects on Fabric having a Pile composed of Shrinkable and Non-shrinkable Yarns
Collins & Aikman Corpn.
BP 855,433 (U.S.A. 13 May 1957)
The back of the fabric is heated so as to cause contraction of the shrinkable yarn in the pile in a predetermined pattern while leaving the non-shrinkable yarn unaffected. C.O.C.

Bibliography on Drying (I p. 170)
Coating and Drying Viscous Liquids on Flexible Webs (I p. 170)
Studies on Textile Finishing Agents. XVIII—Effects of Additives in the Polymerisation and Condensation of *N*-Methylolacrylamide within Cotton Fabrics (III p. 171)
Cyanoethylation of Cotton (VI p. 180)
Behaviour of Wool during Drying (VI p. 182)
Heating Orlon Acrylic Fibre to Render it Fireproof—Fiber AF (VI p. 182)
Schreiner Calendering of Nylon Tricot (VI p. 182)
Surface Treatment of Polyesters (VI p. 182)
Diphenyl Ether Derivatives in Condensation Polymers (XIII p. 189)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Biological Chemistry of Cellulose
J. A. Gascoigne J.S.D.C., **77** (Feb 1961) 53–57

Relationship between Writing Paper and Ink—Comment on the Suggested A.T.I.P. Standard for Measuring Sizing *A.T.I.P. Bull.*, (2) (1960) 84–91
The method, developed with the co-operation of makers and consumers of inks, is a "ruling" method using a range of inks for calibration purposes. The inks consist of solutions of two dyes, Acid Blue VS and Phthalocyanine, the amount of the former determining the aggressiveness of the ink, the latter acting as colorant. The width of the line is 0·6 mm whatever the basis wt. and the thickness of the paper. The method makes it possible to relate paper and ink characteristics and to specify an "average writing paper", thus allowing the matchings of inks to paper. R.A.

A Study of Storing and Preservation Conditions of Wet Spruce Mechanical Pulp. I—Heat Inactivation of Phenylmercuric Acetate as a Preservative for Groundwood Pulp
K. E. Eriksson and H. Kiessling *Svensk Papperstidning*, **63** (15 Nov 1960) 752–757 (*in English*)
The biological activity of PMA is reduced by 60% in the course of one month when the pulp is stored at 37°C, and decreases to almost zero within a few days when stored at 60 or 80°C. Biological activity is only found when the fungicide is reversibly bound to the carboxylic acid groups. R.A.

Brightening of Groundwood
D. F. Manchester, J. W. McKinney, and A. A. Pataky *Svensk Papperstidning*, **63** (31 Oct 1960) 699–706 (*in English*)
New methods for bleaching groundwood are studied and fundamental explanations given for any brightening effects achieved. Spruce groundwood improved in brightness after vapour-phase acetylation, but chemical pulps did not. Methylation with diazomethane was beneficial. R.A.

Degradation of Cellulose by Alkali Cooking
K. Christofferson and O. Samuelson *Svensk Papperstidning*, **63** (31 Oct 1960) 729–732 (*in English*)
The carboxyl content of cellulose rises during alkali cooking. The number of carboxyl groups per mol. is well below unity and decreases slightly with decrease in $(DP)_n$. The number of glucose residues going into soln. for each breakage of the chain is independent of $(DP)_n$, being approx. 40 at 170°C and slightly less at 120°C, indicating that the reaction mechanism is the same at both temp. 19 References. R.A.

PATENTS

Coloured Paper
ICI *BP* 860,937 (5 Apr 1957)
Water-insoluble pigments, together with a water- or acid-soluble condensate of (*a*) an aliphatic aldehyde, (*b*) an ammonium salt or a salt of an aliphatic amine or polyamine, and (*c*) dicyanamide, guanidine, guanylurea, biguanide, monocyclic aromatic primary amines, and/or the crude product containing biuret, guanyl urea, cyanuric acid and guanidine, obtained by heating urea, e.g. at 160°C, are incorporated into the paper during its manufacture. Use of these condensates results in better colour value being obtained than if the same weight of Al sulphate is used. C.O.C.

Antistatic Photographic Films
General Aniline *BP* 861,134 (U.S.A. 7 June 1956)
The silver halide emulsion layer is coated with a compound of formula $R{\cdot}O{\cdot}(CH_2CH_2{\cdot}O)_nH$ (R = Alk of 6–18 C; $n > 3$), e.g. condensate of lauryl alcohol and 12 mol. of ethylene oxide. C.O.C.

Action of Oxidising Agents on Cellulose and Hydrocellulose (VI p. 181)
Effect of Hypochlorite Bleaching Variables on Properties of Prehydrolysed Sulphate Hardwood Pulp (VII p. 183)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Effect of Preparation and Type of Dye on Leather Dyeing
V. Pektor and J. Ondráček
Veda a Vyzcum v Prumyslu Kozedelnem, **5** (1958) 73–100
J. Soc. Leather Trades Chem., **44** (Oct 1960) 484
Leathers of a wide variety of tannages and retannages were dyed at pH 3·0, 4·5, and 6·0 with each of the dyes Chrome Leather Brown 3GN, Alizarine Chrome Red G for Leather, Koregal Blue GG, and Koranil Brown ER.

Information is given on levelness, intensity, exhaustion, effect of electrolytes, and fastness to water, fat-liquor, and light. M.T.

Oils, Waxes, and their Derivatives in Leather Finishing
B. A. Roll *Leather & Shoes*, **139** (No. 4) (1960) 21
J. Soc. Leather Trades Chem., **44** (Dec 1960) 574

Oils and their derivatives are used extensively in leather finishing because of their plasticising, wetting, dispersing, emulsifying, and lubricating properties. Few of the film-forming materials used in leather finishing are sufficiently elastic to withstand the natural movement of the leather surface unless a plasticiser (usually castor oil) is added. Recently monoglycerides of fatty acids have been used as plasticisers. Softer waxes produce a particular surface feel, and the harder provide also a high gloss. Carnauba wax is widely used. M.T.

Polymerisates for Leather Finishing
V. Pektor and J. Ondráček
Kozarstvi, **9** (1959) 109–110, 140–141, 173–175, 203–207
J. Soc. Leather Trades Chem., **44** (Oct 1960) 484

Review, including details of finishing processes and analytical methods of testing leather finishes, results of qualitative and quantitative analyses being described. M.T.

Synthetic Resins in Leather Finishes
R. Shaw *Leather & Shoes*, **139** (No. 4) (1960) 26
J. Soc. Leather Trades Chem., **44** (Dec 1960) 574

Review of finishing methods for upholstery and shoe side leathers, and of changes resulting from introduction of synthetic resins. M.T.

Neutral Salt-free Basic Chromium Salts and Use of Anion Exchangers for their Preparation (III p. 171)
Investigation and Quality Evaluation of Aqueous Protein Leather Finishes (XIV p. 191)

XIII—RUBBER; RESINS; PLASTICS

Polymerisation
F. H. Bolton *Ind. Eng. Chem.*, **53** (Jan 1961) 79–81

A brief but detailed review of research published in 1959 covering processes of polymerisation including ionic, free-radical, condensation and suspension polymerisation, catalytic processes, and block, graft, and radiation-induced polymerisation. 131 References. F.J.

Diphenyl Ether Derivatives in Condensation Polymers
J. D. Doedens and H. P. Cordts
Ind. Eng. Chem., **53** (Jan 1961) 59–62

Chloromethylated diphenyl ethers (I) may be produced by the reaction of HCl and formaldehyde on diphenyl ether. (I), which may contain 16–17% chlorine, can form a condensation polymer on treatment with a Friedel-Crafts catalyst and heating to 100–120°C. The resultant thermosetting foam is flame resistant, tougher, and less friable than equiv. phenolic foams. Polymerisation under pressure or in vacuum yields a thermosetting resin suitable as a binder for inorganic materials. Flame-resistant properties of these materials are discussed, together with their industrial application, although no mention is made of possible uses as flame-retardants in textiles. F.J.

Influence of Wavelengths of Light on the Discoloration of Rubber
A. G. Buswell *Trans. I.R.I.*, **36** (Dec 1960) 263–267

Three white rubber compounds—A, containing no antioxidant; B, containing a non-staining type of phenolic antioxidant; and C, containing an amine staining-type antioxidant, were exposed to varying band-widths of the solar spectrum for up to 75 days. Discoloration in sunlight appears to be due to ultraviolet radiation in the cases of A and B, and partly to visible and partly to ultraviolet radiation in the case of C. W.R.M.

Fibre Resin Preform Moulding
A. Lewin *Trans. Plastics Inst.*, **28** (Dec 1960) 224–232

Preforms of fibre–resin compositions containing cellulose fibres and thermosetting resins are defined and described, and the preparation of preforms is outlined. The processing of fibres, incorporation of resins, and the treatment of the preforms before moulding are discussed. Moulding and finishing techniques are outlined, the economics of the process discussed, and some applications given. W.R.M.

Some Phenolic Resins Soluble in Vegetable Oils. II—Isoamyl Phenol–Formaldehyde Resins
I. Nanu and I. Manovici
Studii și Cercetări, Științe Chimice (Timișoara), **VII** (Jan–June 1960) 169–177

Isoamyl alcohol is used instead of butyl phenol (cf. J.S.D.C., **76** (Nov 1960) 654) to prepare resins sol. in vegetable oils and which may be used in films. H.H.H.

Half a Century of Plastics
H. Saechtling *Kunststoffe*, **50** (Dec 1960) 653–662
German Plastics Digest, **50** (Dec 1960) (12) 1–5 (*condensed English translation*)

Historical. C.O.C.

Acrylic Polymers. IX—Further Investigations on Thermal Changes in Polyacrylonitriles
J. Schurz, A. Ullrich, and H. Zah
Mh. Chem., **91** (Nov 1960) 741–748

Experiments are described on the polymerisation of acrylonitrile and on its condensation with ammonia. An osmometric and viscosimetric study of the thermal changes produced in polyacrylonitrile is also reported, in which an increase in mol. wt. has been established. This is ascribed to further cross-linking, a view supported by rapid diminution in solubility during the thermal change. H.H.H.

PATENTS

Rendering α-Olefin Polymers Adherent to Inks, Coatings, etc.
Montecatini Societa Generale per l'Industria Mineraria e Chimica *BP* 858,623 (Italy 26 June 1958)

Articles made from isotactic polypropylene (isotactic content < 70%) are treated with a liquid of formula—$Ar(COOAlk)_{1-3}$ or $Alk(COOAr)_{3-2}$, e.g. 2-ethylhexyl-phthalate at 140–180°C, long enough for a thin layer of the polymer to be dissolved. They are then treated with a solvent for the ester but not for the polymer, so as to precipitate into the surface as a very fine powder a thin layer of the polymer dissolved by the ester. C.O.C.

Embossing and Printing Polyethylene Film
Wellington Print Works *BP* 860,895 (U.S.A. 13 Aug 1956)

Polyethylene film is heated to soften it and then embossed while simultaneously printing the hollows of the embossed design. C.O.C.

Curing of Polyepoxides
Devoe & Raynolds Co. *BP* 855,205 (U.S.A. 5 Oct 1956)

Use of a neutral quaternary salt as the curing agent for polyepoxides yields compositions which are stable at room temperature or slightly above. C.O.C.

Embossed Plastic Sheets
Konrad Hornschuch *BP* 861,061 (Germany 1 Aug 1957)

Imitation leather for upholstery is obtained by applying parallel strips of plastic to a base sheet and passing the assembly through an embossing roller which imparts a different design to the strips than it does to the uncovered portions of the base sheet.

BP 861,062 (Germany 2 Aug 1957)

The base sheet has panels not strips of plastic applied to it before the embossing. C.O.C.

Emulsifying Agents for Resins (III p. 172)
Rutile Titanium Dioxide (C.I. Pigment White 6) for Pigmenting Laminates containing Aminoplast Resins (IV p. 179)
Surface Treatment of Polyesters (VI p. 182)
Synthetic Resins in Leather Finishes (XII this page)

XIV—ANALYSIS; TESTING; APPARATUS

Determination of Carboxyl Groups
K. Christofferson and O. Samuelson
Svensk Papperstidning, **63** (15 Nov 1960) 749–751 (*in English*)

Hydrolysis with 0·02-M H_2SO_4 at 90–105°C followed by treatment with 7% NaOH at 100°C for 1½–14 h (Samuelson

and Wennerblom *ibid.*, **58** (1955) 713) gives extremely good reproducibility with samples stable in alkaline soln. This method gives results slightly higher than those of the common methods owing to incomplete reaction in the latter, but it can be used only with cellulose containing no, or only small amounts of, carbonyl groups. 10 References.
R.A.

Modified Method for Determination of Active Agent and Degree of Substitution in Carboxymethylcellulose (CMC)
K. Wilson *Svensk Papperstidning*, **63** (31 Oct 1960) 714–715 (*in English*)

The earlier method (*ibid.*, **59** (1956) 218), using pretreatment with 1-N HCl followed by neutralisation with alcoholic NaOH, gives erroneous results with CMC at D.S. $> 0{\cdot}8$, when the CMC swells so that part of the acid remains unneutralised inside the swollen CMC. This is overcome by neutralising the acid with aq. NaOH, when the sample dissolves. Pure CMC is then pptd. with alcohol. The modified method is reproducible and gives correct results with all grades of CMC. R.A.

Detection of *p*-Cresol
F. Feigl and V. Anger *Anal. Chem.*, **33** (Jan 1961) 89–90

The test depends upon the formation of a blue colour lake with magnesium oxide, after coupling with diazotised *p*-nitroaniline. The following compounds also give the test— resorcinol, 1-naphthol, chromotropic acid, H-acid, malonamide, and pyrrole. P.B.S.

Energy-recording Spectrofluorimeter
W. Slavin, R. W. Mooney, and D. T. Palumbo *J. Opt. Soc. Amer.*, **51** (Jan 1961) 93–97

The design of an instrument for the measurement of excitation, fluorescence, and reflectance spectra is described. Advantage is taken of the linear energy response of a thermocouple throughout the wavelength spectrum to record excitation spectra in energy units. A constant energy spectrum from the excitation monochromator is obtained and used to adjust the electrical gain of the fluorescence system, which allows fluorescence spectra to be displayed in energy units. Typical excitation and fluorescence spectra are shown and other applications are indicated. E.COATES

Paper Chromatography in the Identification of 1:2 Metal-complex Dyes and Fluorescent Brightening Agents
P. Miró *Investigación e Información Textil*, **3** (Oct–Dec 1960) 295–302

Using ascending chromatograms, R_f values for eight solvent mixtures are given for 1:2 metal-complex dyes and for fluorescent brightening agents. Detection on the fibre is discussed. S.R.C.

Measuring Complexing Affinities of Aromatic Tanning Agents and Dyes for Chromium (III) Salts
G. Reich, S. Walther, and F. Stather *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, (15) (1959) 50–53
J. Amer. Leather Chem. Assocn., **55** (Nov 1960) 660

The method of Otto (*J. Amer. Leather Chem. Assocn.*, **54** (1959) 477) has been modified. Increasing amounts of dye or tanning agent are mixed with identical amounts of $Cr(NO_3)_3$ soln., NaOH is added to precipitate $Cr(OH)_3$, the solutions are boiled and filtered, and the filtrate is examined for dye (visually) or tanning agent (Cd acetate and pyridine). The amount of dye or agent that can be complexed by the fixed amount of Cr is the largest amount that is completely coprecipitated. Effects of structure on complex-forming capacity of dyes, and effects of masking agents, e.g. phthalate, resorcinolate, in inhibiting complex formation, were studied. M.T.

Analytical Study of the Natural Red Food Dye Alkannin (C.I. 75530)
C. Dušínský and A. Szokolay *Nahrung*, **4** (1960) 356–364
Chem. Abs., **54** (25 Nov 1960) 25346

Alkannin was obtained from the roots of *Alkanna tinctoria* by extraction with petroleum ether; its absorption in the visible and ultraviolet regions of the spectrum was determined. Investigations were made into its polarographic estimation and its identification by paper chromatography in presence of other food dyes [Bixin (C.I. 75120), Curcumin (C.I. 75300), Chlorophyll (C.I. 75810)], and by circular surface chromatography.
C.O.C.

Analysis of Hair Dyes
C. J. Turi *Rend. ist. super. sanità*, **19** (6) (1956) 461–469
Chem. Abs., **54** (25 Nov 1960) 25594

Describes a paper-chromatographic method of identifying the organic compounds in commercial hair tints. The R_f values in butanol, acetic acid, and water are given of *p*-phenylenediamine (C.I. Oxidation Base 10), *p*-aminophenol (C.I. Oxidation Base 6), pyrogallol (C.I. Oxidation Base 32), and resorcinol (C.I. Oxidation Base 31). Numerous colour reactions have been studied to differentiate between these five oxidation bases.
C.O.C.

Status of Colour and Gloss Measurements in Industry
M. P. Morse *Off. Dig. Fed. Soc. Paint Technol.*, **32** (Dec 1960) 1618–1626

Current practices and problems of colour matching and gloss rating by visual and instrumental measurements are described and discussed. Studies by groups from the Federation of Societies for Paint Technology and the ASTM to establish standardised conditions for rating colour and gloss are described and problems encountered in preparing and maintaining permanent colour and gloss standards are outlined. Types of colour and gloss measuring instruments used in industry are briefly described and some methods of translating measurement data to visual appearance are given. Results of ASTM study of colour-difference measurements by commonly used spectrophotometers and colorimeters are evaluated. Comments are made on the advantages and disadvantages of instrumental colour and gloss measurements as compared to visual ratings. J.W.D.

Identification of Individual Nylon, Terylene, and Acrilan Fibres in Admixture
B. Milligan and T. A. Pressley *J. Textile Inst.*, **52** (Jan 1961) T 47

The sample, usually one fibre, is treated with a drop of a 0·05% soln. of Carmoisine W (C.I. Acid Red 14) in 90% formic acid. Nylon loses strength immediately and dissolves in 1–2 min. Remaining fibres are removed, rinsed, and examined. Acrilan fibres are heavily stained, but Terylene fibres are unaffected. Almost any acid dye may be used. M.T.

Possibilities and Limits of the Determination of Fastness Properties of Dyeings
E. Brunnschweiler *Teinture et Apprêts*, **60** (Oct 1960) 143–156

General discussion of the problems arising from widespread use of different methods of testing fastness properties of coloured fibres, with reference to differences in method of testing, evaluation of results, and application of results to practice. Light-fastness testing is first discussed, illustrating how the result depends on numerous variables, e.g. source of light, humidity (variations found in fastness of certain basic and azoic dyes with humidity changes), temperature (Xenotest fades at 55 ± 5°C, Fadeometer at 63 ± 3°C), presence of gases (Basic orange 22, C.I. 48040 fades differently in pure O_2, air, and pure N_2), depth of shade under test, substrate, dyeing procedure (e.g. presence or absence of 2% tetrasodium pyrophosphate affects fastness of Direct Yellow 39 on cotton), pre- and after-treatments (e.g. Direct Blue 86, C.I. 74180, shows different fastness on cotton according to whether the fabric is not finished, is finished with a melamine resin without subsequent washing, or is finished with a urea–HCHO resin with washing). Also discussed are fastness to washing, sea water, perspiration, solvents, and dry cleaning, and the application of fastness figures in practice, i.e. what fastness properties indicate suitability of a dye for a given purpose (cf. Quality Certificate Bureau Ltd. (London), Courtaulds' Tested Quality; Indanthrene and Felisol Associations and A.I.S. recommendations). A.T.P.

Comparison of Instruments for Light- and Weather-exposure Tests
W. Bubser *Melliand Textilber.*, **41** (Nov 1960) 1413–1416

The characteristic features of various test instruments (Xenotest, Weather-Ometer, Brillant-Gerät) with artificial

light sources for light- and weather-exposure testing are reviewed. Comparative tests illustrate the different results given by these instruments. I.G.L.

Microscopic Investigation of Dyed Wool
R. Lassé *Textil-Rund.*, **16** (Jan 1961) 1–3

Describes the testing of the penetrating and levelling agent Neovadin AL in the application of Cibacron dyes to wool by microscopic examination of a yarn cross-section. Details of the preparation of very thin (8–12 μ) cross sections, and of their microphotography (in black and white) are given. P.B.S.

Evaluation of Creasing Due to Laundering of Wash-and-Wear Cotton Fabrics
F. Nestelberger *Textil Praxis*, **15** (Nov 1960) 1161–1168

The author compares and contrasts different laboratory methods described in the literature for the estimation of the degree of creasing of wash-and-wear fabrics which are not ironed after washing and drying (drip or tumbler drying). In agreement with some other workers the author concludes that the wet creasing angle alone is not a sufficient criterion for qualitative assessment of the efficiency of wash-and-wear finishes. The Monsanto wash test in conjunction with three-dimensional wash-and-wear standards enables realistic assessments to be carried out, but relatively large (60 × 90 cm) test samples and wide margin for subjective judgment present certain drawbacks. The author ends by describing his procedure for measuring the creasing angle after washing and drying. With the aid of empirically derived curves the results can be correlated with the Monsanto wash-and-wear standards. In the past few years several thousand fabrics have been successfully tested by this method. L.A.T.

Testing Resin-finished Fabrics
P. Vogel *Melliand Textilber.*, **41** (Nov 1960) 1416–1418

A review of the standard methods employed for testing resin-finished cellulosic fabrics, the accuracy and reliability of the results obtained, and the potential usefulness of these test methods. M.T.

Variation in Wet Crease Recovery Tested by Monsanto and T.B.L. Methods
J. T. Marsh *Text. Research J.*, **31** (Jan 1961) 68

Tests by the Monsanto method have shown little difference between dry and wet crease recovery of untreated cottons, although it is well known that recovery of wet cotton is almost negligible and always very much lower than the dry recovery. Because of this it is unwise to measure both dry and wet recovery by this test for one and the same sample. C.O.C.

Comparison of Standards for Assessing Wrinkling of Fabrics
Z. M. Sudnik *Text. Research J.*, **31** (Jan 1961) 78–79

The methods devised in the laboratories of British Nylon Spinners (*J. Textile Inst.*, **51** (1960) T 227; J.S.D.C., **76** (Aug 1960) 524) and Monsanto, respectively, have been compared. The Monsanto standards are shown to be less critical than those of British Nylon Spinners. C.O.C.

Testing the Properties of Flame-proofed Fabrics
H. J. Reese *Melliand Textilber.*, **41** (Dec 1960) 1559–1563

Flame-proofed fabrics must often fulfil other requirements. The testing of the following finishes is considered and a survey of the different methods in use is given— water-repellent, water-proof, bacteria- and fungi-resistant, rot-resistant. Fabric properties which may be affected by the finish must be checked. They are— air permeability, dimensional stability, weight, colour fastness, and physical properties. W.M.

Finishing of Textiles with Preservatives and the Testing of the Resistance to Cellulose-attacking Microbes. IV— Suggested Standard Method
W. Hausam *Melliand Textilber.*, **41** (Dec 1960) 1563–1569

It is pointed out that results of tests using agar as nutrient substrate may be misleading, since agar favours too much the growth of special micro-organisms (*Aspergillus niger*), suppressing thereby the growth of others. Soil is suggested instead as nutrient substrate, as used in rotting tests. Experiments to corroborate this view are described. W.M.

Quantitative Testing of Water Repellency of Textiles DIN-53888
Faserforsch. und Textiltech., **11** (Nov 1960) 551–553

A stretched circular sample of material is sprayed with water under standard conditions. The size and shape of the drops formed on the material are noted and the water absorption expressed by—

$$W = \frac{G_s - G_tr}{G_tr} \times 100\%$$

where G_{tr} and G_s are the weight of the sample before and after spraying. W.R.M.

Testing Water-repellency of Yarns
H. Modlich *Melliand Textilber.*, **41** (Nov 1960) 1418–1421

A new method is described which permits the water-repellency of fibres and yarns (particularly of man-made fibres) to be determined prior to their use in the manufacture of umbrella cloths. Full details of the method are given, and results obtained are discussed. I.G.L.

Investigation and Quality Evaluation of Aqueous Protein Leather Finishes
F. Stather and H. Herfeld
Ges. Abhandl. deut. Lederinst. Freiberg/Sa., (15) (1959) 145–156
J. Amer. Leather Chem. Assocn., **56** (Jan 1961) 45

Methods are outlined for determining solids; water; volatile matter; ash; pigment; pigment ash; ammonia; protein; plasticiser; pH; rate of sedimentation; particle size; clumping tendency; viscosity; acid stability; and hiding power. Data are given for 40 widely varying finishes of different colours. It is concluded that rigid specifications for a finish cannot be set up; at most, analysis may serve to compare successive deliveries of the same finish. M.T.

Paper Chromatography of Precondensates and Products of Hydrolysis of Synthetic Resins
K. Thurnher *Melliand Textilber.*, **41** (Nov 1960) 1421–1426

A method is described of chromatographic analysis of precondensates and the products of hydrolysis of synthetic resins (derivatives of similar character to many textile finishing agents) which includes a new technique for rendering the various bands visibly detectable. Details of the method are given, the band-developing process consisting in spraying the paper with Tollens Reagent (1 vol 0·1-N silver nitrate : 1 vol N caustic soda : 1 vol 5-N aq. ammonia). The chromatographic detection of resin precondensates is described, and the results of experiments with various solvent mixtures are given, demonstrating the dependence of R_f value on solvent composition and on the polar nature of the precondensate molecule, the latter being, in turn, dependent on the number of hydroxy groups. Hydrolysis of resins was effected under various reaction conditions, the products of hydrolysis in each case being analysed chromatographically. I.G.L.

Detection of End-groups in Polyacrylonitrile by Differential Infrared Spectroscopy
I. Matsubara
Bull. Chem. Soc. Japan, **33** (Nov 1960) 1624–1626

Differential infrared spectroscopy, previously reported useful for the identification and quantitative estimation of minor constituents in organic substances, has been applied to the detection of end-groups in polyacrylonitrile. Differential analyses have been carried out with success in combination with the potassium bromide disc technique. Results of analysis of samples of differing degrees of polymerisation (polymerised using ammonium persulphate–sodium bisulphite redox system as initiator) are given, and compared with those obtained with samples for which other initiators (azo-bis-isobutyronitrile, ammonium persulphate, and Fe^{2+}–sodium bisulphite redox system) had been used. It is also reported that quantitative estimations of the end-groups by the differential method, using polymers of known end-group content, showed the limit of detectability of both sulphate and sulphonate end-groups by this method to be ca. 0·002 mmol/g polymer. I.G.L.

Dipole Moments and Sorption Characteristics at the Solution–Solid Interface of a Series of Substituted Azobenzenes (IV p. 172)

Effect of Weathering on the Properties of Textile Fibres— III (VI p. 180)

Polymerisates for Leather Finishing (XII p. 189)

XV— MISCELLANEOUS

The Nature and Origin of Colours

Jac van Essen *Die Farbe*, **9** (Sept 1960) 31–47

The dispersion of white light into the spectrum colours and the additive mixing of colours are discussed and lead to a historical summary of the theories of colour vision. Some physiological aspects of the problem are dealt with, from the construction and operation of the eye to differences in vision in different animals. The possible effects of light of different wavelengths on the retina, giving rise to photodegradation of pigments and the subsequent propagation of electrical impulses along the nerve fibres, are considered. Variations in the individual colour vision of observers are stressed and the importance of psychological influence is noted. The interconnections between the response from each of a pair of eyes and the interpretation at cortical level of response at retinal level, with the added complexities of colour adaptivity and experience, are brought into this discussion. The overall picture presented is the scientific background to a metaphysical theme. E. COATES

Opponent Chromatic Induction; Experimental Evaluation and Theoretical Account

D. Jameson and L. M. Hurvich *J. Opt. Soc. Amer.*, **51** (Jan 1961) 46–53

An experimental arrangement is described whereby stimulus mixtures were provided by the use of two projectors with means for independent control of chromaticity and luminance by coloured and neutral filters. Five square areas, together forming a cross, could be projected onto a diffusing translucent screen, each area subtending approximately 3° at the eye (observer 110 cm from the screen). The focal area was the left-hand square limb of the cross and could be considered alone in a dark surround or together with one or more other squares (inducing areas). Forty observers obtained matches, by a glance technique, with black-masked Munsell samples, shielded from the main optical path and illuminated by a standard tungsten light source (2800°K; 90 ft-c). Five individuals made matches for the same stimulus conditions and the Munsell notations were transformed to tristimulus values, which were averaged. These average X, Y, Z values were transformed to the chromatic response values of the opponent colours scheme, details for which are given. Changes in the focal pattern by the induction patterns are expressed in the same terms. It is shown that the induced chromatic responses are opponent to, but proportional in magnitude to, the mean chromatic activities of the inducing field for given degrees of contiguity. The function has a negative slope, e.g. in the red–green system, as the redness of the inducing area is increased the focal area appears greener (less red). Mutual interactions in a variegated pattern of surround stimuli tend to counterbalance the effect of increasing the total area of surround field. E. COATES

Grammar School Chemistry

J. Roy. Inst. Chem., **85** (Jan 1961) 1–2

A report recently published for the Science Masters' Association indicates that grammar school provisions for the teaching of chemistry are inadequate as regards available space, equipment, and staff. Most teachers spend time carrying out tasks proper to a laboratory technician. Too few girls are studying chemistry (which means, in turn, that there will be fewer science teachers) and their schools have even poorer facilities than boys' schools. Much higher expenditure is needed. J.W.D.

Suggested Changes in the Chemistry Syllabus

E. J. Rothery *J. Roy. Inst. Chem.*, **85** (Jan 1961) 2–4

Teaching of elementary chemistry should, as in certain European countries, be revised and obsolete concepts given much less attention than at present. Modern theories should be introduced at an early stage; this contention is supported by discussion of several subjects— wave-mechanics and quantum theory, electronegativity, oxidation and reduction, acid–base theory, and the chemistry of non-aqueous soln. Chemistry should be approached through a knowledge of atomic structure and electron transfer; this gives unity to the subject and, also, bridges the gap between elementary physics and chemistry. J.W.D.

British Chemical Industry— I

C. J. Thomas *J. Roy. Inst. Chem.*, **85** (Jan 1961) 5–12

A survey of employee distribution, diversity of products, economics, organisational structure, and expenditure on research. J.W.D.

A New Method for Producing Coloured Cements

P. I. Bozhenov and L. I. Kholopova *Tsement.*, **26** (4) (1960) 15–20

Chem. Abs., **54** (25 Nov 1960) 25681

Small amounts (> 1%) of one of a number of metal oxides are added to the mix of an iron-free Portland cement. The resulting colour is not caused by formation of new coloured compounds, e.g. chromates, manganates and ferrites, but rather to change in colour of the basic clinker materials with the entrance of coloured oxides into the crystal lattice of the mineral, or by formation of solid solutions. The colours of many natural minerals are formed in this way and are characterised by great variety and high intensity, even at very low concentrations of the coloured medium. C.O.C.

Plant Maintenance and Working Conditions— II

F. H. Slade *Text. Manuf.*, **87** (Jan 1961) 3–7

Successful processing is often dependent upon the provision of adequate and accurate air conditions. The use and maintenance of the following units are discussed: condensers, conditioning plant, air washers, heater batteries, air circulation, clean air, and automatic dampers and control gear. S.B.D.

PATENTS

Chromic Acid Treatment of Feathers

U.S. Secretary of the Army *USP* 2,928,713 (23 Apr 1957)

Feathers are given permanent curliness, water repellency, and resistance to moths by thoroughly cleansing them, treating with Na_3PO_4 to modify their cystine linkage, and then treating for 2 h in an aqueous solution of chromic acid, basic chromic sulphate, or a mixture of hydrazine sulphate and spent chromic acid. C.O.C.

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U.S. Secretary of the Army *USP* 2,928,714 (23 Apr 1957)

Feathers are rendered resistant to moth and microbiological attack and given permanent curliness and water-repellency by thoroughly cleansing them, and then treating first with Na_3PO_4 and then with an aqueous solution of an epichlorohydrin, e.g. 2·5% aq. 1-chloro-2,3-epoxypropane for 150 min at 45°C. C.O.C.

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Dipole Moments and Sorption Characteristics at the Solution–Solid Interface of a Series of Substituted Azobenzenes (IV p. 172)

Manufacturing Problems of Zinc Chromates (C.I. 77955) for Use as Corrosion-inhibiting Pigments (IV p. 175)

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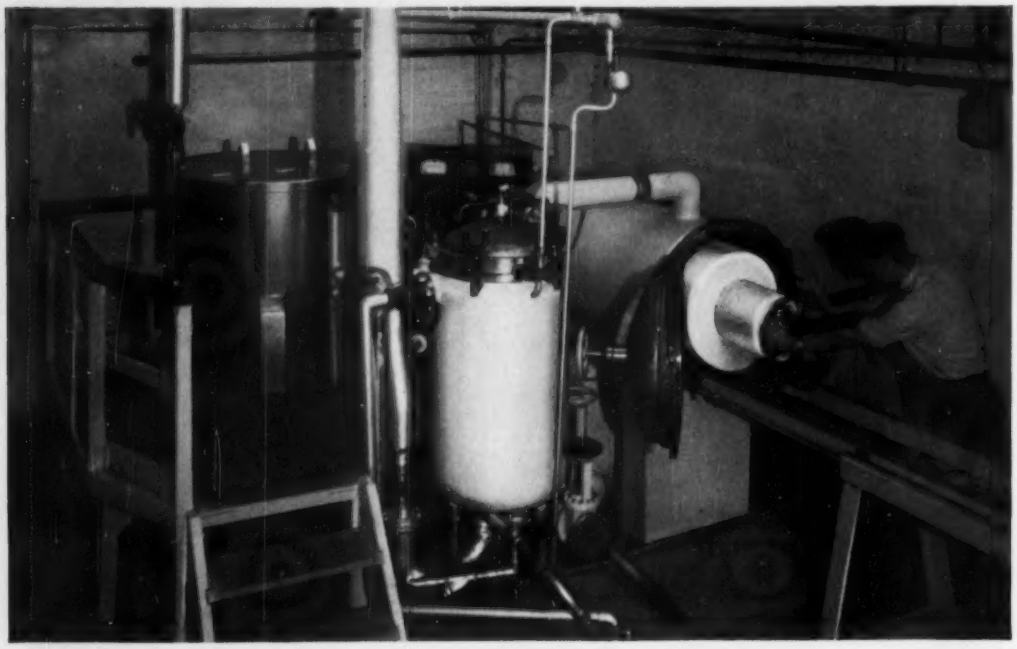

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continued overleaf

APPOINTMENTS etc—*continued*

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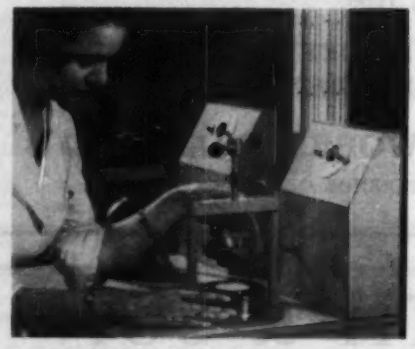

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Advance Announcement

THE NINTH LONDON LECTURE

will be given at
THE WALDORF HOTEL
LONDON WC2
on
Friday 20 October 1961
at
7 pm
by
Timothy O'Brien Esq
(*Head of Design, A.B.C. Television Ltd*)
on
"COLOUR IN THE THEATRE"

The Society of Dyers and Colourists

Standard Methods for the Determination of the Colour Fastness of Textiles

The methods of fastness testing sponsored by the Society of Dyers and Colourists are published as a separate handbook under the title "Standard Methods for the Determination of the Colour Fastness of Textiles", the first edition appearing in 1955.

Developments in fastness testing, particularly internationally, necessitated the publication in 1958 of a supplement to the handbook and since then further developments have occurred. **Instead of issuing a second supplement, however, the Society's Fastness Tests Co-ordinating Committee decided that it would be more convenient for the Society to issue a Second Edition of Standard Methods for the Determination of the Colour Fastness of Textiles** which is now available from the offices of the Society.

The Second Edition contains the tests, etc., which appeared in the *Journal* as Sponsored Publications since the supplement was published—

Light: Daylight at High Humidity
The use of fading lamps
Pleating and Setting

and also three tests which are now appearing for the first time—

Light: Daylight (U.S.A. Method)
Xenon lamp
Vulcanising: Hot air

Mainly in the interest of international agreement two of the original tests have been substantially modified: these are—

Burnt-gas fumes
Perspiration

whilst minor modifications have been made to certain others.

Many of the tests in the handbook have become Recommendations of the International Organisation for Standardisation and have been adopted as National Standards in many countries including the United Kingdom. A list of equivalent national standards is included in each test which should be of value where exports and imports are concerned.

To facilitate ease of reference, the tests have now been arranged alphabetically and a standard lay-out has been adopted.

THIS HANDBOOK IS UNDOUBTEDLY THE MOST COMPREHENSIVE AND UP-TO-DATE SOURCE OF INFORMATION ON COLOUR FASTNESS TESTING IN THE WORLD AND IS INDISPENSABLE WHEREVER COLOUR FASTNESS TESTS ARE MADE.

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THE SOCIETY OF DYERS AND COLOURISTS

Dean House 19 Piccadilly Bradford Yorkshire

The price is

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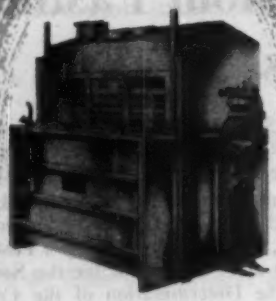

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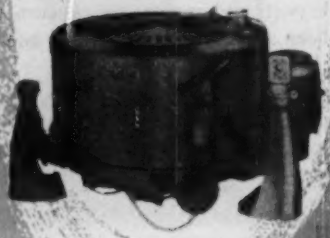

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